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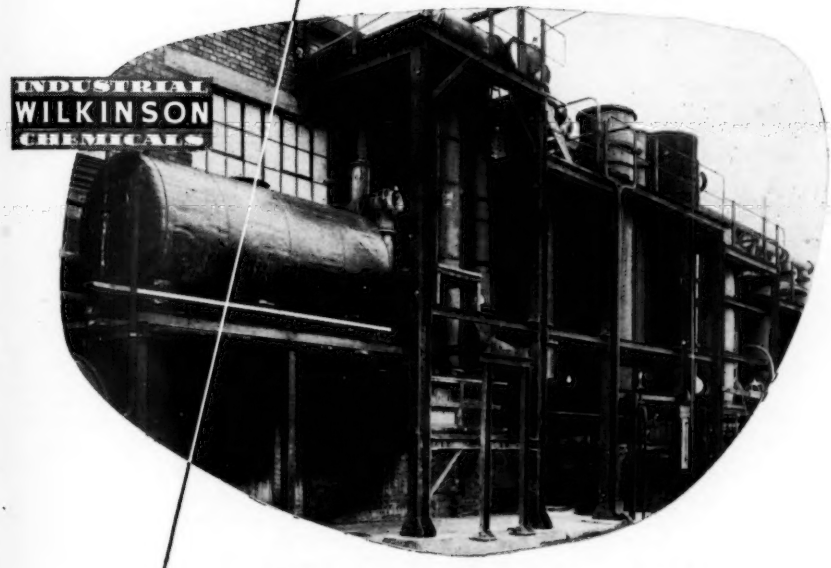

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

2910. **The technique of qualitative X-ray analysis.** W. Epprecht (*Chimia*, 1954, **8**, 165–175).—In this review of X-ray diffraction by powders, the Debye - Scherrer and Guinier techniques are compared, identification of pure substances is described, and methods of analysis of mixtures are briefly discussed. A. B. DENSHAM

2911. **Titrations in non-aqueous solutions. VIII. Some reductimetric titrations in glacial acetic acid.** O. Tomíček, A. Stodolová and M. Heřman (*Chem. Listy*, 1953, **47** [4], 516–520).—The following potentiometric titrations were carried out in glacial acetic acid against the S.C.E.: Br, CrO_3, $KMnO_4$, ICl, BrO_3', IO_3' and chloramine-T with 0·05 *N* $Na_2S_2O_4$; Pb tetra-acetate, CrO_3, $KMnO_4$, BrO_3' with 0·05 *N* vanadyl acetate; Br, Pb tetra-acetate and ICl with 0·1 *N* $AsCl_3$ and 0·1 *N* catechol. The course of the titrations with $Na_2S_2O_4$ and vanadyl acetate is discussed in detail and titration curves are given. The reducing power of the agents changes with the acidity of the medium under the influence of an added strong acid or base. $SnCl_2$ is unstable in acetic acid. G. GLASER

2912. **Titrations in non-aqueous solutions. IX. Neutralisation titrations in anhydrous formic acid.** O. Tomíček and P. Vidner (*Chem. Listy*, 1953, **47** [4], 521–525).—The course of the following acid - base titrations in 98 to 98·8 per cent. formic acid is described: Na formate, urea, pyridine, sulphapyridine, betaine, 8-hydroxyquinoline, aniline, ephedrine and caffeine with 0·1 *M* $HClO_4$, and $HClO_4$ with 0·1 *M* Na formate. For potentiometric titrations a quinhydrone or hydrogen electrode was used; visual titrations were carried out with gentian violet, neutral violet, safranine, malachite green and orange I as indicators. Removal of water from formic acid (repeated distillation and freezing out) is troublesome and formic acid is therefore a less convenient titration medium than glacial acetic acid. G. GLASER

2913. **Titrations in non-aqueous solutions. X. Neutralisation titrations in anhydrous pyridine.** O. Tomíček and S. Křepelka (*Chem. Listy*, 1953, **47** [4], 526–530).—Acid - base titrations in anhydrous pyridine of $HClO_4$, formic acid, benzoic acid and the ion-exchange resin Zeokarb with *N* piperidine, *N* diethanolamine soln. and 0·5 *N* NH_3 soln. were carried out with a glass electrode for potentiometric and bromothymol blue as indicator for visual titrations. A hydrogen electrode was used to determine potentials of pyridine solutions of $HClO_4$, formic acid, piperidine and diethanolamine. The soln. of NH_3 in pyridine was prepared by adding $NaNH_2$ to the aq. base. G. GLASER

2914. **Ultra-micro method of chemical analysis. III. Potentiometric titration.** I. P. Alimarin and M. N. Petrikova (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 127–133).—Potentiometric titrations on $\simeq$ 0·001 ml of solution are described. The solution is contained in a horizontal capillary vessel having a fused-in platinum wire as working electrode. A capillary containing KCl and connected to a calomel electrode, a capillary micro-burette, and a capillary tube for introduction of gas to stir the solution, are dipped into the solution, the whole being observed under a microscope. Amounts of the order of 10^{-6} to 10^{-7} g can be determined with an error of 2 to 3 per cent. The apparatus is illustrated and examples of its use, *e.g.*, for determination of Fe, Cr and V, are given. G. S. SMITH

2915. **Use of ultra-violet rays in analytical chemistry. Cerimetry in ultra-violet rays.** K. P. Stolyarov (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 141–149).—The end-point in many titrations with ceric sulphate is determined by use of radiation of 365 mμ and visual observation of a suitably disposed fluorescent screen. With one drop of the titrant in excess the screen goes dark. The method is applied to the determination of Sb, Sn, Sr, Ca, Cd, Ag, Pb, As, oxalates, sulphites, arsenites, thiosulphates, thiocyanates and iodides, and to the assay of Pb and Sn in lead alloys. G. S. SMITH

2916. **Separations by simple counter-current techniques.** A. Brunzell and H. Hellberg (*Ann. Pharm. Franç.*, 1954, **12** [4], 296–307).—The separation of substances, between a mobile and stationary phase in simple decantation vessels, is considered in terms of the volumes of the phases and the partition "ratio" (not the classical coeff.), *viz.*, the ratio of total concn. in the two phases, which also remains nearly constant. Partition "ratios" are established for a number of barbiturates and other drugs between the pairs of phases; 0·5 *M* aq. Na_2CO_3 - ether, sat. aq. $NaHCO_3$ - ether, 0·5 *M* aq. Na_2CO_3 - $CHCl_3$, sat. aq. $NaHCO_3$ - $CHCl_3$, 0·5 *M* aq. HCl - $CHCl_3$, and 0·5 *M* aq. HCl - ether. Partition pH curves between $CHCl_3$ and phosphate buffers for a number of drugs are established, so that conditions for a particular separation can be chosen in advance. A number of detailed examples of separations are given. E. J. H. BIRCH

2917. **Acetylation of paper for chromatography.** J. E. Scott and L. Goldberg (*Chem. & Ind.*, 1954, [2], 48–49).—One part of paper was kept overnight in pyridine (previously stored over NaOH), then steeped in a mixture of 16 parts of pyridine and 10 parts of acetic anhydride for 9 days at 37° C, air-dried for 24 hours, and then extracted with ether and methanol. The paper had a white to pale yellow colour, did not absorb water readily and had good mechanical properties. The amount of combined acetic acid was constant at 25 per cent. for several batches; this could be increased after reaction at a higher temperature for a longer time. D. LIFF

2918. **The acid dew-point.** J. R. Rylands and J. R. Jenkinson (*J. Inst. Fuel*, 1954, **27**, 299–309).—Earlier attempts to determine acid dew-point temp. by electrical "dew-point meters" are briefly reviewed, and attention is drawn to the unreliability of the results. The acid dew-point temp. varies only slightly with wide variations of SO_3-content in combustion gases. The mechanisms of H_2SO_4 deposition on surfaces at temp. less than acid dew-point temp., and of mist formation are investigated by a technique not dependent on electrical measurements. A controlled stream of hot air containing a known amount of acid and water vapour is passed through a glass U-tube cooled to a definite temp. Any acid collected on the surface is subsequently washed off and determined volumetrically. Possible serious corrosion effects of HCl in combustion gases at temp. below the water dew-point temp. are suggested. D. R. GLASSON

2919. **Extraction of metal thiocyanate complexes with tri-*n*-butyl phosphate.** L. M. Melnick (*Dissert. Abstr.*, 1954, **14** [5], 760–761).—The extraction of metal thiocyanates in aq. solution with tri-*n*-butyl phosphate has been investigated, together with the controlling factors and the analytical applications. Extraction of ferric thiocyanate is best with the following: (*i*) a min. thiocyanate to Fe ratio of 5 to 1 for max. extraction; (*ii*) 25 ml of tributyl phosphate as solvent (Fe may be extracted also with tributyl phosphate - CCl_4 mixtures, the vol. of tributyl phosphate being equal to or in excess of that of CCl_4); and (*iii*) pH range 0·85 to 1·98 (the Fe fraction extracted decreases with increasing ionic strength). The fraction of Fe extracted increases with a decrease in temp., the temp. coeff. of extraction being −0·3 per cent. per 1° C between 13° and 58° C, and extraction of Fe is independent of concn. for concn. of Fe between 0·04068 and 0·4068 *M*. Cobalt^{II} and cupric thiocyanates are quant. extracted from aq. solution with Fe, thus permitting determination of Ni in their presence in excess. Molybdenum^{V} thiocyanate in H_2SO_4 is extracted with tributyl phosphate, thus affording a simple method for determining Mo in steel. For extraction of cupric thiocyanate: (*i*) the min. thiocyanate to Cu ratio for max. extraction is between 23 and 24 to 1; (*ii*) 20 ml of tributyl phosphate is the optimum solvent extraction vol.; (*iii*) extraction is independent of pH over the range 2·58 to 5·13; (*iv*) the fraction of Cu extracted increases with a decrease in temp., extraction being complete at 18·5° C and the temp. coeff. of extraction being − 0·9 per cent. per 1° C between 18·5° and 55° C; and (*v*) extraction increases with increasing concn. of Cu. L. F. TAYLOR

2920. **A method of metallurgical microspectroscopy.** F. R. Bryan and C. H. Neven (*Metal Progress*, 1953, **64** [6], 82–85).—A method for identification of microconstituents of metallurgical specimens is proposed. In drilling the specimen, the spindle speed should be less than 3000 r.p.m. and CCl_4 should be used as lubricant and coolant. Chips are recovered by flowing drops of flexible collodion (10 per cent in butyl acetate) over them and stripping off; the sample (as little as 0·1 μg) is placed on the end of a spectrographically pure graphite electrode and moistened with a drop of butyl acetate for improved adhesion. In arcing (2 amp.), a total exposure of 10 sec. is adequate, spectra being taken on No. 1 plates or film, or, for greater sensitivity, on Eastman Type 103–0. Inclusions can be identified, a series of closely spaced samplings can provide evidence of segregation and segregates can be identified, metallic constituents of individual crystals can be identified if the structure is not too fine, and underlying layers of multiple platings can be sampled and identified individually by drilling into their cross sections. By means of a series of drillings, diffusion characteristics can sometimes be determined. LIGHT MET. BULL. ABSTR.

See also Abstract 2999.

2.—INORGANIC ANALYSIS

2921. **Precision concentration analysis of D_2O/H_2O by means of phase-contrast refractometry.** E. Inglestam, E. Djurle and L. Johansson (*J. Opt. Soc. Amer.*, 1954, **44** [6], 472–477).—Inglestam's phase-contrast refractometer (*Ark. Fys.*, 1953, **6**, 287) enables optical path lengths to be determined with a precision of the order of $\lambda/1000$, and by its use the refractivity function of a mixture of heavy and light water can be determined. Various factors influencing the measurements are discussed and it is concluded that D_2O contents of mixtures may be determined to within about 0·002 mole per cent. at all concentrations. B. S. COOPER

2922. **Spectrographic determination of sodium and potassium in coal ashes.** C. H. Anderson and C. D. Beatty (*Anal. Chem.*, 1954, **26** [8], 1369–1371).—A coal ash sample (1 part) (<200 mesh) containing 0·3 to 5 per cent. of Na or K is well mixed with buffer - internal standard mixture (either 4 parts of Li_2CO_3 containing 3·2 per cent. of Rb_2CO_3 or 6 parts of $BaCO_3$ containing 1·5 per cent. of Li_2CO_3 and 6 per cent. of K_2SO_4 for K and Na determinations, respectively.) Ten mg of the final mixture are subjected to an electric arc under standard conditions and the determination is completed in the standard spectrographic manner, by use of the intensity ratios: Na 3303·0 to Li 3232·6 and Na 3302·3 to Li 3232·6 for Na, and K 4044·1 to Rb 4215·6 and K 4047·2 to Rb 4215·6 for K determinations. Time requirements and precision (4·1 per cent. error for K and 4·3 per cent. for Na) are claimed as being considerably better than for existing methods. Cyanogen and iron do not interfere. D. A. PANTONY

2923. **The qualitative detection of potassium, rubidium and caesium by wet methods: a critical survey.** W. Geilmann and W. Gebauhr (*Z. anal. Chem.*, 1954, **142** [4], 241–254).—An extensive examination, including the use of radioactive isotopes in some instances, of the available methods for the detection and separation of the alkali metals showed that their reliability leaves much to be desired. Many are shown to be particularly unreliable for the analysis of mixtures of salts of the alkali metals, where the concentration of one is much greater than that of the others. Suitable methods are recommended and their sensitivities are given. It is possible to detect traces of Cs and Ru in Na or K, but difficult to detect K in presence of large amounts of Cs and Ru. P. S. STROSS

2924. **New analytical procedures for copper.** C. Abrisqueta Herrera (*Anales Univ. Murcia*, 1952–3, **11**, 363–417).—Procedures involving the formation of $CuHg(SCN)_4$ are given for the determination of Cu in pure samples and in the presence of large quantities of H_2SO_4, acetic acid, H_3PO_4, HNO_3 and Fe^{+++}, Al^{+++}, Cr^{+++} and Mn^{++}. Chloride ion interferes.

For pure samples or those containing acetic acid or H_3PO_4, add 10 ml of a 10 per cent. glycerol soln., heat to 40° C and add, very slowly with constant stirring, at least 20 ml of a soln. containing 10 per cent. of $Hg(SCN)_2.2NH_4SCN$(**I**) and 0·1 per cent. excess of NH_4SCN. Cool below 20° C and filter through sintered glass. Wash 5 times with 10 ml of 2 per cent. **I**, 3 times with 3 ml of 0·25 per cent. **I** and once with water. Dry over $CaCl_2$ and then at 105° C. If H_2SO_4 is present use 20 per cent. instead of 10 per cent. glycerol. If HNO_3 is present neutralise the excess of acid with 40 per cent. Na acetate before proceeding as above. If $Fe^{...}$ is present convert it to a complex by adding H_3PO_4 before pptg., and adding 5 per cent. v/v of H_3PO_4 to the first 5 washes. Alternative procedures are given to be followed in the presence of large amounts of $Cr^{...}$, $Al^{...}$ or $Mn^{..}$. It is also possible to determine Cu volumetrically by dissolving the $CuHg(SCN)_4$ in KI and titrating the liberated I with $Na_2S_2O_3$. CHEM. ABSTR.

2925. **Use of complexones in chemical analysis. XL. Reductimetric determination of silver.** R. Přibil, J. Doležal and V. Simon (*Chem. Listy*, 1953, **47** [7], 1017–1022).—Silver is determined potentiometrically and polarographically at pH 4 to 6·5 (at which the redox potential of the system FeY''/FeY' is $0·117_2$ V) with $FeSO_4$ in the presence of complexone III (**I**). *Procedure*—To 5 to 100 mg of Ag, add 0·1 *M* **I** in an amount corresponding to a Ag to **I** ratio of 1 to 1. Adjust the pH to 5 with an acetate buffer (5 to 10 ml), dil. to a $Ag^{·}$ concn. of 0·005 *M* and titrate with 0·1 *M* $FeSO_4$. Up to 200 mg of Pb, 50 mg of Cu, 50 mg of Bi and 50 mg of Cd do not interfere, provided sufficient **I** is added to bind these metals. G. GLASER

2926. **Indirect absorptiometric determination of magnesium with 4-aminophenazone (4-aminoantipyrin).** R. A. McAllister (*Analyst*, 1954, **79**, 522–523).—In the method proposed Mg is pptd. from ammoniacal soln. with 8-hydroxyquinoline. The ppt. is washed and redissolved in dil. HCl and the 8-hydroxyquinoline is determined colorimetrically by means of the orange-red colour formed when oxine is coupled with 4-aminophenazone and the complex is oxidised with $K_3Fe(CN)_6$ in alkaline soln. The sample should contain up to 50 μg of Mg, the final vol. of the soln. being 50 ml. The optical density is measured on a Spekker absorptiometer with an Ilford 602 or Chance OB1 filter. The calibration graph, which is linear, is prepared similarly from standard amounts of Mg.
A. O. JONES

2927. **Direct volumetric determination of magnesium in calcareous minerals.** R. E. Muraca and M. T. Reitz (*Chemist Analyst*, 1954, **43** [3], 73–74).—The proposed procedure gives a method for rapidly and directly determining Mg in presence of a large excess of Ca. A 0·2-g sample is decomposed in a platinum crucible with HF, H_2SO_4, and, finally, $K_2S_2O_7$. After cooling, the product is dissolved in 100 ml of water with 5 ml of HCl, boiled and cooled, and 20 g of mannitol are added. The Mg is precipitated with 5 *M* KOH, and, after filtration and washing, is dissolved again in dilute HCl; mannitol is added, and the Mg is reprecipitated with KOH. The ppt. is dissolved in a buffer soln. of 3 g of NH_4Cl, 15 ml of conc. aq. NH_3 and 25 ml of H_2O, diluted with water and titrated with disodium ethylenediaminetetra-acetate, with Eriochrome black T as indicator. H. P. PAGET

2928. **The determination of magnesium and zinc in aluminium alloys by high vacuum distillation.** P. Urech and R. Sulzberger (*Aluminium*, 1954, **30** [4], 163–164).—A simpler form of apparatus is proposed for the routine determination of Mg and Zn by the method of Urech *et al.* (*Brit. Abstr. C*, 1949, 323). The vacuum is produced by a two-stage rotary oil pump. Aluminium alloys are heated to 800° C, at which temp. Mg is distilled quant. in 90 min.; Mg or magnesium alloys are heated to 650° C. The amount of Mg is determined by weighing the sample before and after distillation, a correction being made for Zn, which is also removed quantitatively. The absolute error is ± 0·01 per cent. of Mg and the method is applicable to aluminium alloys containing from 0·1 per cent. of Mg upwards. Nineteen samples can be simultaneously distilled in ≃ 150 min. and four batches can be dealt with in a day. B. J. W.

2929. **A spectrophotometric micro-titration of calcium.** R. A. Chalmers (*Analyst*, 1954, **79**, 519–521).—A modification is described of the spectrophotometric method and apparatus of Sweetser *et al.* (*Brit. Abstr. C*, 1953, 306) for detecting the end-point in titrations with disodium ethylenediaminetetra-acetate (**I**), and the method is applied to the micro-titration of Ca. The cell carrier of a spectrophotometer is replaced by a Perspex cover painted black and fitted with a slot carrying a Perspex cell and a polythene stirrer. The cell contains ≯ 1 ml of a Ca soln. (10 to 20 μg of Ca), and murexide powder is added and 2 *N* NaOH until a pink colour appears. After addition of a few more drops of NaOH, the soln. is diluted to ≃ 3·5 ml and is then titrated with 0·01 *N* **I**, the optical density at 610 mμ being determined after each addition of titrant. When the hitherto increasing optical density becomes constant for three readings, the values obtained are plotted against vol. of titrant added; the intersection of the graph with the straight line representing the final constant value indicates the end point.
A. O. JONES

2930. **Phosphate interference in the flame-photometric determination of calcium.** L. Leyton (*Analyst*, 1954, **79**, 497–500).—Emission of Ca in the air - acetylene flame from soln. of $CaCl_2$ decreases linearly with increasing phosphate content until the P to Ca ratio is 1 to 2, Ca emission being afterwards independent of the phosphate concn. The influence of up to 100 p.p.m. of K and up to 50 p.p.m. of Na is negligible. The interference by phosphate is attributed to $Ca_3(PO_4)_2$, which is excited only slightly in the air - acetylene flame and hardly at all in the coal-gas flame. With an air - acetylene flame, Ca determinations can be corrected for phosphate interference if the phosphate content is known, or, in presence of excess of phosphate, it can be made directly if a calibration graph for $Ca_3(PO_4)_2$ is available. With a coal-gas flame, phosphate must be eliminated. This can be effected by adsorption of the Ca on a cation-exchange resin (Zeocarb 215) with subsequent elution (Mason, *Brit. Abstr. C*, 1953, 135). A. O. JONES

2931. **A study of the separation of zinc from certain other elements by means of anion exchange.** C. C. Miller and J. A. Hunter (*Analyst*, 1954, **79**, 483–492).—Five to fifty mg of Zn in 2 *N* HCl soln. containing ≯ 100 mg of metal ions can be quant. adsorbed on the strong-base anion-exchange resin Amberlite IRA-400(Cl). After percolation of 2 *N* HCl to a total of 50 ml, the eluate contains almost

all the Al, Mg, Cu, Co, Ni, Mn^{II}, Cr^{III}, Fe^{III}, Th, Zr, Ti^{IV}, U^{VI}, Be and Ca. Cd, Sn^{IV}, Sb^{III} and Bi and some Pb and In are held with the Zn. Zn, Cd and In, 20 per cent. of the Sn and small amounts of Sb, Bi and Pb are eluted with water and 0·025 *N* HNO_3. Two procedures for separating the Zn in the eluate are described; both depend upon formation of the 8-hydroxyquinoline complex, and the one in which pptn. is made in an alkaline tartrate medium is generally applicable. With alloys containing 2·5 to 34 per cent. of Zn, results have been promising and precision moderate. The quant. separation of 0·5 mg of Zn from 100 mg of Al or Mg has been achieved. A. O. JONES

2932. **Colorimetric determination of zinc and copper with 2-carboxy-2′-hydroxy-5′-sulphoformazylbenzene.** R. M. Rush and J. H. Yoe (*Anal. Chem.*, 1954, **26** [8], 1345–1348).—Aliquots (10 ml) of a soln. containing 25 to 100 μg of Zn^{++} and Cu^{++} are treated as follows. *Zinc determination*—The soln. is buffered to pH 9 and the reagent [0·13 per cent. 2-carboxy-2′-hydroxy-5′-sulphoformazylbenzene (Zincon) containing 2 ml of *M* NaOH per 100 ml] (3 ml) is added. After making up to 50 ml the light absorption is measured at 620 mμ against a reagent blank. Zn^{++} concn. is deduced from calibration curves. *Copper determination*—This is similar to that of Zn^{++}, except that the soln. is buffered to pH 5·2 and the absorption is measured at 600 mμ. Another procedure based on cation exchange (Dowex 1 in conjunction with *M* or 0·01 *M* HCl) avoids the interferences of Cu^{++}, Fe^{+++} (> 500 μg), Co^{++} and Ni^{++} (> 1000 μg). The Beer-Lambert law holds over the concn. examined, and a sensitivity of 1 pt. per 7×10^7 is given. D. A. PANTONY

2933. **Influence of complex-forming ions on the analytical behaviour of mercury.** F. Burriel-Martí and S. Bolle-Taccheo (*Inf. Quim. Anal.*, 1954, **8** [3], 77–85).—Theoretical expressions are derived for the redox potential of Hg in presence of various ions that remove Hg^{++} from solution. The effect of such ions is to reduce the Hg - Hg^{++} potential eventually to negative values, proportionately to the concn. of the complexing ion and in order of efficiency: $Cl' < Br' < I' < SCN' < CN'$. This explains the known increase in reducing power of Hg in presence of these ions (*cf. Anal. Abstr.*, 1954, **1**, 1520). D. P. YOUNG

2934. **The determination of traces of boron with quinalizarin.** E. A. Johnson and M. J. Toogood (*Analyst*, 1954, **79**, 493–496).—Quinalizarin dissolves in conc. H_2SO_4 to give an intense violet-blue soln., the colour of which changes through violet and mauve to red with increasing additions of water. A soln. in 93 per cent. H_2SO_4 has a colour intermediate between the blue and the red. With addition of 1 ml of water to 10 ml of this soln. the colour changes towards red, but if the water contains borate the change is less, and, with sufficient boron, the change is towards the blue. With fixed conditions and a reagent of the same composition, the relation between colour change and boron content is linear. Investigation of the reaction showed that the acid concn. is of importance for the sensitivity and range when this change is used as a colorimetric or spectrophotometric method for determining boron. It was found also that boron may be determined by titrating with water the coloured soln. formed in its presence until the colour is equal to that of the same reagent without boron, the amount of boron present being ascertained from a calibration graph. Hence a spectrophotometer and colour standards are not required in application of the method. A. O. JONES

2935. **Determination of indium in aluminium bronze alloys by flame photometry.** V. W. Meloche, J. B. Ramsay, D. J. Mack and T. V. Philip (*Anal. Chem.*, 1954, **26** [8], 1387–1388).—The alloy (0·1 g) containing 0·5 to 2·5 per cent. of In is dissolved in the minimum of HNO_3, the soln. is boiled and diluted to 100 ml, and the In is determined flame-photometrically (oxy-hydrogen flame) at 451·1 mμ by comparison with a linear calibration curve derived from standards containing 0 to 100 p.p.m. of In plus 900 p.p.m. of Cu and 50 p.p.m. of Al. Sensitivity is 0·2 p.p.m. and mean deviation is ± 0·1 per cent. By using the Cu and Al in the standards, interference due to Cu and Al are avoided, and Zn, HNO_3 and HCl at concn. up to *N* do not interfere, but *N* H_2SO_4 depresses flame intensity. D. A. PANTONY

2936. **Separation of rare earths and their accompanying elements. VIII. New colorimetric determination of cerium.** Z. Hagiwara (*Technol. Rep. Tôhoku Univ.*, 1953, **18** [1], 16–39).—Ce^{IV} in concn. of 5 to 35 mg per litre forms a red-brown cupferron complex that can be extracted from a solution in 0·15 *N* acid with *iso*pentyl or *n*-butyl acetate and determined by a photo-electric photometer with a blue filter. Ca, Pr, Nd, Sm, Gd, Er, Yb, and Y give white ppt., but do not interfere with the extraction. Th, Mg, Ca, Mn, F′, and PO_4''' do not interfere, whilst Fe^{III} gives a colour much less intense than that due to Ce^{IV}. A. R. PEARSON

2937. **Gas analysis absorbents for carbon monoxide.** Y. Okinaka and T. Kato (*Technol. Rep. Tôhoku Univ.*, 1953, **18** [1], 12–15).—The absorptive powers for CO of ammoniacal and acid (HCl) solutions of Cu_2Cl_2 have been compared. The most efficient, containing Cu_2Cl_2 (11·5 g), H_2O (50 ml), and conc. aq. NH_3 (43 ml), had an equilibrium p[CO] of 0·003 atm. after absorbing 0·714 vol. of CO. The addition of ethanolamine (**I**) reduces the v.p. of the solution, and a solution containing Cu_2Cl_2 (35 g), $CuCl_2$ (3·5 g), **I** (36 g), aq. NH_3 (30 ml), aq. HCl (sp.gr. 1·18) (15 ml) and H_2O to 100 ml is recommended. A. R. PEARSON

2938. **Determination of moisture in sodium bicarbonate. Karl Fischer method.** L. N. Gard and R. C. Butler (*Anal. Chem.*, 1954, **26** [8], 1367–1368).—Moisture in 100 g of $NaHCO_3$ is extracted by shaking for 1 min. with 130 ml of methanol. After 5 min., the suspension is filtered and the H_2O plus $NaHCO_3$ in 50 ml of the filtrate is determined by a standard Karl Fischer procedure. The dissolved $NaHCO_3$ is determined separately on the remaining 50 ml of filtrate by titration with 0·1 *N* HCl (methyl purple). After allowance for a blank value and the dissolved $NaHCO_3$, the H_2O is calculated with a precision of ± 0·005 in 0·01 to 0·16 per cent. D. A. PANTONY

2939. **Iodine chloride as indicator in the oxidation of thiocyanate by potassium permanganate.** G. S. Deshmukh and M. K. Joshi (*Z. anal. Chem.*, 1954, **142** [4], 275–277).—Oxidation of thiocyanate to sulphate with $KMnO_4$ is normally only 95 per cent. complete. Iodine chloride acts as a catalyst and indicator (in the presence of carbon tetrachloride). The HCl concentration is critical and must be between 1·5 and 2 *N*. The end-point

is indicated by the disappearance of the brown colour in the CCl_4 layer. The method, which is particularly suitable for the estimation of metals that form stable thiocyanates, is suggested for the indirect estimation of cerium. P. S. STROSS

2940. **Determination of thiocyanate by ceric sulphate.** G. S. Deshmukh and M. K. Joshi (*J. Indian Chem. Soc.*, 1954, **31** [5], 413–414).—Thiocyanate can be oxidised quantitatively by $Ce(SO_4)_2$ according to the equation—

$$6Ce(SO_4)_2 + KCNS + 4H_2O = 3Ce_2(SO_4)_3 + KHSO_4 + 3H_2SO_4 + HCN.$$

The KCNS solution is added to an excess of standard $Ce(SO_4)_2$ solution, and then conc. H_2SO_4 is added until the acidity is $\simeq 6\ N$. The solution is boiled for 10 to 15 min. until no more HCN is evolved. After cooling the soln. to room temp., the excess of $Ce(SO_4)_2$ is estimated with standard ferrous ammonium sulphate solution by means of ferroin indicator. The results are reproducible and differ from the results obtained on parallel estimations by the Volhard method by $\simeq 0{\cdot}2$ per cent. J. H. WATON

2941. **Note on the rapid determination of silicon in steels.** R. Desguin and R. Boulin (*Chim. Anal.*, 1954, **36** [9], 245–246).—The method of Gotô and Kakita (*Brit. Abstr. C*, 1951, 164) is described, and comparative results for its use are given. The drillings are dissolved in HNO_3, the solution is diluted to $\simeq 8$ ml (3 to 4 *N* in HNO_3), and 10 ml of 40 per cent. HF and 6 g of KCl are added in a polystyrene vessel. The pptd. K_2SiF_6 is filtered, washed with KCl solution and titrated with NaOH. E. J. H. BIRCH

2942. **Methods for the analysis of iron and steel. XXXI. Silica in blast-furnace slag.** British Standards Institution (B.S. 1121: Part 31: 1954, 6 pp.).—The finely ground sample is fused with Na_2CO_3 in a platinum crucible first at 400° to 500° C for 10 min. and then at 1000° C for 15 min. The fused mass is dissolved in $HClO_4$ and the silica is dehydrated by fuming. The silica is filtered off and the silica in the filtrate is recovered by fuming as before. This recovery from the filtrate can be omitted in routine analyses. The combined silica precipitates are ignited at 1050° to 1100° C for 30 min., cooled and weighed. The residue is then fumed with HF and H_2SO_4 and the silica is determined by the loss in wt. The method covers the range of silica contents found in blast-furnace slags; reproducibility is within $\pm$ 0·15 per cent. This procedure is recommended as a referee method. C. J. KEATTCH

2943. **Spectrophotometric and polarographic determinations of soluble silicate.** M. A. DeSesa and L. B. Rogers (*Anal. Chem.*, 1954, **26** [8], 1278–1284).—A u.v. spectrophotometric method, based on the measurement of the yellow molybdosilicic acid colour, for determination of low concn. of SiO_2 in MgO and $MgCO_3$ is described; results are compared with those by a new polarographic procedure for SiO_2. A soln. containing sufficient SiO_2 to bring the final concn. up to 1 to 5 p.p.m. is made up to approx. 75 ml with 5 ml of molybdate reagent (50 g of ammonium molybdate tetrahydrate per litre of water), 2 ml of 60 per cent. $HClO_4$ and water. After allowing the mixture to stand for 30 min., 40 per cent. aq. tartaric acid (4 ml) is added, and the mixed soln. is made up to 100 ml with water. The absorption is measured at 332 mμ after 15 min., and compared with standards prepared similarly. Variation of the final absorption spectrum with pH, molybdate concn. and order of mixing of reagents is discussed in detail. Only interferences from PO_4''', AsO_3''' and SO_4'' are studied; of these the first two are effectively removed by the use of tartaric acid. The polarographic procedure is as follows: the SiO_2 soln. (preferably obtained by solution in aq. NH_3) is treated with 4 *M* aq. NH_4NO_3 (25 ml) to act as supporting electrolyte, ammonium molybdate reagent (5 ml), 9·2 *M* HNO_3 (2 ml), and 0·1 per cent. gelatin (1 ml) to act as maximum suppressor. The soln. is made up to 100 ml and the polarogram is prepared at + 0·23 V by use of an aliquot under nitrogen. D. A. PANTONY

2944. **Photometric determination of titanium with chromotropic acid in pig iron and steels, including chromium nickel steel, with removal of the iron and alloying metals.** W. Koch and H. Ploum (*Arch. Eisenhüttenwesen*, 1953, **24**, 393–396).—Although other photometric determinations of Ti in steel require previous removal of the Fe from the soln. of the sample, the proposed method does not. Quadrivalent Ti gives a red complex with chromotropic acid in acid solutions, but tervalent Fe gives a green compound. As divalent Fe gives colourless solutions, Fe does not need to be removed, but has to be reduced to the divalent state; this can be done by ascorbic acid, the optimum pH value being 2·5. The method can be used for steels with 18 per cent. of Cr and 8 per cent. of Ni, with Ti contents up to 1 per cent., and for pig iron. The solutions used are: 1 g of ascorbic acid in 100 ml of water, the soln. being prepared each day; 6 g of the sodium salt of chromotropic acid dissolved in 100 ml of H_2O, the soln. not being used for 2 days; and acetate buffer soln. containing 100 g of Na acetate dissolved in about 120 ml of H_2O. After addition of 800 ml of acetic acid, the buffer soln. is diluted to 100 ml. The sample (0·5 g) is dissolved in 10 ml of conc. HCl. CHEM. ABSTR.

2945. **Fluorimetric detection and estimation of germanium.** N. Appala Raju and G. Gopala Rao (*Nature*, 1954, **174**, 400).—A germanium soln. (0·1 to 1·0 ml) is added to 3 ml of a soln. of 0·5 to 1·0 g of resacetophenone in 100 ml of glacial acetic acid, to which has been added 6 ml of syrupy H_3PO_4. After adjustment of the vol. to 10 ml with H_3PO_4, the sample is exposed to u.v. light, when a greenish-yellow fluorescence, the intensity of which varies with the Ge concn., confirms unequivocally the presence of Ge. Limit of sensitivity is given as 1 in 10,000, and the limit of identification as 100 μg. NO_2', NO_3', F' and CrO_4'' quench the fluorescence. D. A. PANTONY

2946. **Determination of germanium in coal, coal ash and flue dust.** W. J. Frederick, J. A. White and H. E. Biber (*Anal. Chem.*, 1954, **26** [8], 1328–1330).—A sample of coal, coal ash or flue dust (5 g, 100 mesh) containing $\gtrsim$ 3 mg of Ge is ignited with $CaCO_3$ at 1000° to 1050° C after roasting at 480° C. The cooled mixture is boiled with 200 ml of conc. HCl in presence of 100 ml of water and 50 per cent. aq. K_2CrO_4 (2 ml) in a simple distillation assembly. The distillate (50 ml) is quant. collected under ice-cold water, the $GeCl_4$ being swept over in a stream of CO_2. Two hundred ml of water are added and the soln. is neutralised with aq. NH_3 (methyl red). To this, at 10° to 15° C, are added 25 per cent. aq. NH_4NO_3 (20 ml), 2 per cent. aq. ammonium molybdate (16 ml), 2 *N* HNO_3 (30 ml) and cinchonine reagent (2·5 per cent. in 2·5 *N* HNO_3) (9 ml). The ppt. is isolated after 3 hr. at 0° C, dried at 160° C and weighed. Allowance is

made for a blank determination, and results are based on an empirical factor of 0·02955. Maximum deviations on several experiments are given as ± 0·04 mg. By the use of Bi as internal standard a spectrographic method has been devised that is suitable for determination of low concn. of Ge; however, coal gives erratic results.

D. A. PANTONY

2947. **A volumetric procedure for the determination of zirconium in its binary alloys with uranium.** G. W. C. Milner and P. J. Phennah (*Analyst*, 1954, **79**, 475–482).—After separation, Zr in acid soln. is treated with 0·1 *M* ethylenediaminetetra-acetic acid soln. (**I**) in slight excess of the amount required to form a complex. Cresol red is added and then aq. NH_3 to the yellow point of the indicator. Ammonium acetate is added and the pH is adjusted to between 5 and 6. The soln. is boiled, cooled, salicylic acid is added as indicator and the excess of **I** is back titrated with standard 0·1 *M* $FeCl_3$ soln. The preliminary separation of Zr from other metals with organic precipitants is discussed. For preliminary separation in the rapid analysis of Zr - U binary alloys, the Zr is pptd. as the mandelate from a soln. of the alloy in $HClO_4$. A. O. JONES

2948. **Quantitative determination of tin by means of arsonic acids.** A. I. Portnov (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 175–178).—Tin is quant. pptd. by 4-hydroxy-3-nitrophenylarsonic acid in 1 per cent. HCl. Cu, Zn, Pb and most other elements do not interfere. After being washed with water, the ppt. is dissolved in HCl, KBr is added followed by excess of 0·1 *N* $KBrO_3$, then, after 15 min., excess of 0·1 *N* arsenite is added and the excess of arsenite is titrated with $KBrO_3$.

G. S. SMITH

2949. **Determination of lead in metallurgical products.** J. Kinnunen and B. Wennerstrand (*Chemist Analyst*, 1954, **43** [3], 65–67).—The method described makes the use of $PbSO_4$, which is slightly soluble, unnecessary and depends on the formation of complexes with diethyldithiocarbamate. One g of brass or bronze is dissolved in 8 ml of aq. HNO_3 (1 + 1); 5 ml of 20 per cent. Rochelle salt soln., 10 ml of conc. aq. NH_3, 30 ml of 20 per cent. KCN soln. and 10 ml of 2 per cent. Na diethyldithiocarbamate soln. are added. The solution is extracted with chloroform (20 ml in all), and the extract is treated with 5 ml of HNO_3; the chloroform is then driven off. Two ml of Rochelle salt solution are added, the excess of acid is neutralised with aq. NH_3, and 10 ml of a buffer solution are added (54 g of NH_4Cl and 350 ml of aq. NH_3 in 1 litre). After dilution to 125 ml, 3 ml of KCN solution are added and the solution is titrated with 0·01 *N* Na ethylenediaminetetra-acetate, with Eriochrome black T as indicator. Alternatively, the extraction may be carried out with 30 ml of ethyl acetate, the extract being washed with 50 ml of H_2O and the Pb returned to the aq. phase on shaking with 50 ml of H_2O and 2 ml of slightly acid 1 per cent. $CuSO_4$ soln The titration is carried out as before. Interference by Mn can be minimised by adding 4 g of KCN before the titration. Procedures are also given for determination of Pb in lead concentrates and in barium ores by similar principles.

H. P. PAGET

2950. **Analysis of lead titanate.** J. H. van der Meulen (*Verfkroniek*, 1954, **27** [4], 94–95).—Water is determined by drying at 120° to 125° C, and free PbO by extraction with boiling 0·25 *N* acetic acid and pptn. as $PbCrO_4$. Total Pb and Ti are determined by a new procedure. One g of sample is mixed successively with 9 g of ammonium persulphate, 10 ml of conc. H_2SO_4 and 1 ml of water. After being heated for 20 to 30 min., the mixture is cooled, 25 ml of cold water are added, followed by 50 ml of hot water, and the mixture is heated to boiling. The pptd. $PbSO_4$ is filtered off and weighed. The Ti in the filtrate is pptd. as hydrated TiO_2 by means of ammonium carbonate, and weighed as TiO_2 or titrated after reduction in a Jones reductor. The results are expressed as percentages of free PbO, PbO and TiO_2 combined as $PbTiO_3$, and free TiO_2. D. R. DUNCAN

2951. **Estimation of thorium by organic reagents. II. 2 : 4-Dichlorophenoxyacetic and guaiacoxyacetic acids.** S. K. Datta and G. Banerjee (*J. Indian Chem. Soc.*, 1954, **31** [5], 397–401).—Thorium is estimated by pptn. with hot 1 per cent. solutions of 2 : 4-dichlorophenoxyacetic acid (**I**) and guaiacoxyacetic acid (**II**). When **I** is used, the Th solution is neutralised with NaOH soln. so that the pH is > 2·8. After the addition of the reagent, a white flocculent ppt. collects at the bottom of the beaker if the solution is warmed a little. When **II** is used, the thorium soln. is made just neutral to Congo red so that the pH is > 4·4. After adding the reagent and boiling the soln. for a few min., a white granular ppt. appears. In both instances the ppt. is filtered on a Whatman No. 42 filter-paper, washed with a hot 0·2 per cent. soln. of the reagent, and ignited to ThO_2. When compared with the wt. of ThO_2 obtained by the oxalic acid method, pptn. with reagent **I** is found to give a max. error of ± 0·2 per cent. and with reagent **II** a max. error of ± 0·6 per cent. These reagents will separate Th from Ca, Sr, Ba, Mg, Zn, Al, Cr, Co, Ni, and, in particular, Ce and La, when a single pptn. is effective for a thoria - rare earth ratio of 1 to 40 for **I** and 1 to 20 for **II**. In this way, Th can be estimated in an acid extract of monazite. Only the ppt. with reagent **I** dries to a salt of definite composition (24·40 per cent. of Th) that can be used for direct weighing. J. H. WATON

2952. **The semi-micro Kjeldahl method for the determination of nitrogen in coal.** A. H. Edwards (*J. Appl. Chem.*, 1954, **4** [6], 330–340).—Semi-micro techniques may be used if the procedure is strictly standardised. Electrical heating is recommended with preliminary experiments to determine the correct rate of heating to give a clearing time of 7 to 10 min. Selenium, although unsatisfactory alone as a catalyst, may be used with either mercuric sulphate or vanadium pentoxide. Blank corrections are reduced by adding a splash head to the Markham distillation apparatus. D. LIFF

2953. **A modified universal semi-micro Kjeldahl method for the determination of nitrogen in coal and other carbonaceous materials.** A. E. Beet (*J. Appl. Chem.*, 1954, **4** [7], 373–379).—The sample size is adjusted to give ≃ 2 mg of nitrogen (0·1 g coal), and 10 ml of conc. sulphuric acid are added. After heating for 5 min. to liquefy the mixture, ≃ 0·05-g lots of potassium permanganate are added, with shaking after each addition until the surface of the acid is covered with a slight froth. Further additions of permanganate are made more cautiously until the colour of the digest changes from dark brown to dark orange, lemon, and finally becomes colourless. This point may be difficult to judge if the material contains more than 1 to 2 per cent. of iron. The

digest is allowed to cool slightly and a further 0·2 g of permanganate is added gradually with shaking, the temperature is then raised just to the boiling point and the digest is then immediately allowed to cool and diluted with ≃ 20 ml of water. The solution is transferred to the distillation apparatus via the reagent funnel with two successive 10-ml lots of water followed by 40 ml of 10 *N* NaOH soln. The distillate is titrated with 0·01 *N* acid.

D. LIFF

2954. **Polarographic determination of hydroxylamine.** A. Blažek (*Chem. Listy*, 1953, **47** [7], 1003–1007).—Hydroxylamine is determined by polarographic titration in two ways: (*i*) with 0·1 *N* $TiCl_3$ in 0·2 *M* oxalic acid (10 ml) at 50° C by use of the dropping-mercury electrode, and (*ii*) with 0·1 *N* $KBrO_3$ in 2 *M* HCl (8 ml) containing 40 per cent. KBr (2 ml) at 60° to 70° C by means of the rotating platinum electrode.

G. GLASER

2955. **Alkali azides in the analytical separation of nitrates and nitrites.** R. Suárez-Acosta (*Inf. Quím. Anal.*, 1954, **8** [4], 115–118).—The rate of destruction of nitrites is increased with the amount of NaN_3 used, reaching an optimum with a relative concn. of NaN_3 to $NaNO_2$ of ≃ 4 to 1. Acidity also increases the rate of destruction of the nitrites. High values are obtained by a Devarda alloy reduction method for the amount of NO_3' present after the NO_2' has been eliminated. Oxidation of nitrite probably occurs during the treatment with NaN_3, particularly when the acidity is high.

L. A. O'NEILL

2956. **Simple method for estimating phosphine in air during the production of zinc phosphide poisonous baits.** W. Filz (*Mitt. Chem. ForschInst. Wirt. Öst.*, 1954, **8**, 61–62).—A simple method of estimating PH_3 in air is to bubble the sample through 0·1 *N* or 0·02 *N* aq. neutral or ammoniacal $AgNO_3$, filter off the silver and titrate the remaining $AgNO_3$ with KCNS. The accuracy is ± 15 per cent. Max. permissible concn. are 0·5 mg per litre for short periods and 0·01 mg per litre for 8 hours.

A. B. DENSHAM

2957. **Potentiometric determination of phosphorus in phosphates.** A. Margara (*Ann. Chim., Roma*, 1954, **44** [5–6], 321–323).—The coarse ppt. of phosphorus as $MgNH_4PO_4$ obtained by carrying out the pptn. in the presence of high concn. of NH_4Cl is insolubilised by treatment with aq. NH_3. The ppt. is filtered and thoroughly washed free of NH_3 to pH 7·85, first with H_2O, then with saturated aq. $MgNH_4PO_4$. The ppt. is then dissolved by treatment with 0·1 *N* HCl (*x* ml):

$$2\,MgNH_4PO_4 + 4HCl \rightarrow MgH_4(PO_4)_2 + 2NH_4Cl + MgCl_2.$$

Excess of HCl is then titrated potentiometrically at glass and calomel electrodes with 0·1 *N* NaOH (*y* ml) to pH 4·2. Then P (in mg) = 1·55 (*x* − *y*).

L. A. O'NEILL

2958. **Analysis of phosphate mixtures by filter-paper chromatography.** J. Crowther (*Anal. Chem.*, 1954, **26** [8], 1383–1386).—Phosphate mixtures containing 60 to 90 μg of P are quantitatively separated on paper by upward flow in single phase solvents. *tert.*-Butanol (80 ml), water (20 ml) and formic acid (5 ml) are used for phosphates containing not more than 4 P atoms, *iso*propanol (80 ml), water (20 ml) and formic acid (5 ml) for not more than 6 P atoms, and *n*-propanol (60 ml), water (20 ml) and aq. NH_3 (20 ml) for up to 9 P atoms per anion. The chromatogram bands are sprayed with molybdate reagent; after warming and then exposure to u.v. for 1 min. easily identified blue colours are produced. The bands are quant. determined as follows: the filter-paper is cut and the blue segments are leached separately with 0·1 *N* aq. NH_3 (25 ml) and a 20-ml aliquot is digested at 100° C for 30 min., cooled and treated with 12·5 per cent. ammonium molybdate (1 ml) and then 0·6 per cent. hydrazine hydrochloride soln. (1 ml). The mixture is diluted to 50 ml and digested at 100° C for 10 min. Absorption is measured at 830 mμ. The P concn. is calculated from standards. Mean deviation of results is given as ± 0·83 per cent., except where streaking of the bands occurs (especially with the propanol - water - formic acid solvent), when deviation rises to 2 per cent. Polyvalent cations cause streaking of the bands, and glycerol and citric acid prevent formation of the blue colour. SiO_2 does not interfere.

D. A. PANTONY

2959. **Radiochromatographic study of the kinetics of exchange adsorption of phosphate ions on inorganic adsorbents.** E. N. Gapon, D. D. Ivanenko and V. V. Rachinskii (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **95** [3], 567–570).—The possibility of studying the distribution of an ion adsorbed on a chromatographic column by measuring the radioactivity of tracer atoms is illustrated by its application to the adsorption of phosphate ions on Na and Ca alumina exchange columns, ^{32}P being used as tracer.

R. C. MURRAY

2960. **Anodic polarographic wave of [quadrivalent] vanadium aged in alkaline solution.** I. M. Kolthoff and P. T. Toren (*Anal. Chem.*, 1954, **26** [8], 1361–1362).—The current - voltage curves at a dropping-mercury electrode of O-free solutions of VO^{++} over the pH range 9·1 to 13·7 were investigated several days after mixing the reagents. Values of $E_{\frac{1}{2}}$ vary from − 0·25 to − 0·42 V and those of the diffusion current constants from − 1·12 to − 0·75 amp. over the same pH range. For fresh solutions, diffusion current constants are between − 1·466 and − 1·41 amp. over a similar pH range. It is suggested that slow association of the VO^{++} occurs in alkaline solution.

D. A. PANTONY

2961. **Separations with a mercury cathode: determination of certain metallic impurities in vanadium salts.** W. E. Schmidt (*Dissert. Abstr.*, 1954, **14** [5], 756).—A procedure based on the electrolytic - polarographic method of Furman, Bricker and McDuffie (*Brit. Abstr. C*, 1950, 351) has been established for determining small amounts (25 μg per g) of Zn, Cd, Fe, Co, Ni, Pb and Cu in conc. vanadium solutions (0·2 to 0·5 *M*). Electrolytic oxidation, or stripping, at a controlled potential, as an alternative to distillation, yielded recoveries of ≃ 0·3 p.p.m. of Cu, Pb, Cd and Zn from amalgams, without introducing detectable amounts of Hg into the solution.

L. F. TAYLOR

2962. **Colorimetric determination of arsenic after volatilisation as arsine.** H. W. Berkhout and G. H. Jongen (*Chemist Analyst*, 1954, **43** [3], 60–61).—Material containing 1 to 25 μg of As is placed in a conical flask with 25 ml of H_2O, 25 ml of hydrochloric acid, 1 ml of 30 per cent. w/v KI soln. and 2 ml of 25 per cent. w/v $SnCl_2$ soln. After 15 min. 10 g of granulated zinc are added, and the flask is connected to an absorption tube containing a 0·5-cm layer of cellulose powder previously treated with $HgBr_2$ soln. After 15 min. the absorption tube is placed in the mouth of a calibrated flask, soaked with 0·5 ml of NaOBr solution for a few

min., and then washed with 1 ml of 2 *N* H_2SO_4 and 5 ml of H_2O. Then 0·4 ml of ammonium molybdate soln., 2·5 per cent. w/v in 2·5 *N* H_2SO_4, and 0·2 ml of 2·5 per cent. w/v hydrazine sulphate soln. are added, and the flask is heated for 5 min. in boiling water. After being made up with water to 10 ml, the transmittance of the solution is measured at 720 mμ, at which Beer's law is obeyed. The colour is stable for at least 1 hr. H. P. PAGET

2963. **Spectrophotometric determination of arsenic, phosphorus and silicon in the presence of each other.** M. A. DeSesa and L. B. Rogers (*Anal. Chem.*, 1954, **26** [8], 1381–1383).—Molybdosilicic, molybdoarsenic and molybdophosphoric acid solutions are prepared as described above (*Anal. Abstr.*, 1954, **1**, 2943) and the absorption is measured at 332 mμ to determine the total Si, As and P. *iso*Pentyl acetate completely extracts molybdophosphoric acid from aq. soln. and the P is determined spectrophotometrically on the organic extract at 330 mμ. It extracts molybdoarsenic acid from 15 per cent. ethanolic soln. only. Arsenic is then determined absorptiometrically on the organic phase at 323 mμ. The Beer - Lambert law holds for all measurements. D. A. PANTONY

2964. **New method for micro-determination of antimony and quantitative separation of stibine.** F. V. Zaykovsky (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 155–161).—Amalgamated Zn and concentrated HCl reduce $Sb^{\cdots}$ quantitatively to SbH_3. The SbH_3 reacts with I in a tube attached to the reaction vessel to give SbI_3, which together with the excess of I is dissolved in KI and tartaric acid; the iodine is reduced by means of ascorbic acid and the solution is heated to give antimonyl tartrate. The Sb is determined by the iodide - thiourea colorimetric method. G. S. SMITH

2965. **Photocolorimetric determination of antimony in ores by the iodide - thiourea method.** D. N. Finkelshteyn and G. N. Kryuchkova (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 150–154).—The use of thiourea as a reducing and complexing agent increases the accuracy and selectivity of the colorimetric iodide method for determining Sb. In its presence, Sb can be determined without interference from Cu, Fe, Sn, As and Hg. A method for determining Sb in ores is described. G. S. SMITH

2966. **Titration of bismuth with ethylenediaminetetra-acetic acid. Spectrophotometric end-points.** A. L. Underwood (*Anal. Chem.*, 1954, **26** [8], 1322–1324).—To a soln. containing 5 to 100 mg of $Bi^{\cdots}$ are added chloroacetic acid (2 g) and aq. *M* $Cu(NO_3)_2$ (1 ml). The vol. is adjusted to approx. 100 ml, and the pH to ≃ 2 with 0·88 aq. NH_3 diluted (1 + 1) or 5 *M* NaOH, in a spectrophotometer titration cell. Absorptions are measured at 745 mμ with successive additions of standard 0·1 *M* disodium ethylenediaminetetra-acetate. Alternatively, 0·5 to 10 mg of $Bi^{\cdots}$ may be determined as follows. Chloroacetic acid (0·5 g) is added to the $Bi^{\cdots}$ soln., the pH is adjusted to approx. 2 at a vol. of 100 ml and the soln. is warmed. On cooling, the absorption is adjusted to zero in a spectrophotometer by slit-width control. Aq. *M* thiourea (5 ml) is added and the titration performed as above. In each case extrapolation of the two linear arms of the graphs of absorption against vol. of titrant gives the end-point for Bi. Interference from Sn, As and Sb are avoided by standard separations, but $Fe^{\cdots}$ and large excesses of $Pb^{\cdot\cdot}$ interfere. Results are given for the analyses of some bismuth alloys. D. A. PANTONY

2967. **A photometric determination of bismuth in lead - zinc alloys.** E. Asmus (*Z. anal. Chem.*, 1954, **142** [4], 255–266).—The proposed method, in which the bismuth - thiourea colour is used, overcomes interference due to other elements commonly present in bismuth-containing alloys, *e.g.*, As, Sb, Cu, Ag, Cd, Zn, Th and Fe, and avoids losses of Bi from co-precipitation. *Procedure*—Dissolve 500 mg of alloy at 70° C in 50 ml of a filtered acid mixture containing 300 g of acetic acid, 200 g of citric acid, 100 ml of HNO_3 (sp.gr. 1·15) and water to 500 ml, adding a few ml of 70 per cent. $HClO_4$ to alloys containing 10 per cent. or more of Sn to speed up solution. Cool to room temp., add 25 ml of HNO_3 (sp. gr. 1·15) and make up to 100 ml with water. Transfer 50 ml of this solution to a second calibrated flask and add, dropwise and with constant shaking, exactly 20 ml of a 20 per cent. w/v thiourea solution. Dilute both solutions to 100 ml with water and, using the first solution as a blank, measure the extinction of the second by the use of a mercury lamp and Hg 436 filter. For alloys containing more than 20 per cent. of Sn, it may be advisable to add a further 25 ml of the above acid mixture to each of the two flasks before making up to the mark. Comparisons of this method with gravimetric methods when applied to standard samples shows that the latter methods are liable to gross errors. The yellow complex is shown to be a compound of one mol. of Bi with three of thiourea. P. S. STROSS

2968. **Polarographic determination of dissolved oxygen in dilute sulphite waste liquor.** H. S. Levine and O. J. Williams (*Anal. Chem.*, 1954, **26** [8], 1297–1302).—In a polarograph cell at constant temp. are placed 5 *M* acetate buffer (120 g of sodium acetate trihydrate and 240 ml of glacial acetic acid made up to 1 litre with water) (0·5 ml) so that the final pH is 4·1, and 0·5 per cent. thymol blue (4 drops). The water sample (50 ml), containing ≯ 3 per cent. of sulphite waste, is added and a polarogram is taken at − 0·6 V. The measurement is repeated after displacement of oxygen by a stream of nitrogen. The current difference is a direct measure of the oxygen concn., which is calculated from a calibration curve prepared by the use of distilled water standardised by the Winkler method. Error is stated to be ± 2·1 per cent. D. A. PANTONY

2969. **Determination of hydrogen sulphide in mineral waters containing quantities not detectable by ordinary methods.** B. M. Colombo and P. C. Federici (*Sperimentale*, 1954, **4** [5–6], 105–109).—The hydrogen sulphide is entrained in CO_2, which is then absorbed in KOH, and the sulphide is titrated iodimetrically. A diagram of the absorption system is given. The application of the method to two natural waters containing $1{\cdot}52 \times 10^{-5}$ and $3{\cdot}69 \times 10^{-5}$ g per litre of the gas is described. R. C. MURRAY

2970. **Rapid determination of the sulphate content in aluminate liquors.** L. Zombory, A. Vigh-Somogyi and T. Pintér (*Acta. Chim. Acad. Sci. Hung.*, 1953, **3**, 519–535).—Three methods are suitable for rapid plant control. (*i*) Dilute 5 ml of the aluminate liquor with 5 ml of H_2O, heat and acidify with HCl until a clear soln. is obtained. Add 4 g of solid NH_4Cl, boil and add 5 ml of 0·5 *N* $BaCl_2$ soln. When the mixture is cool, add 3 ml of ethanol dropwise and then 1 ml of freshly prepared 0·25 per cent. aq. Na rhodizonate indicator soln. Titrate at once with 0·2 *N* Na_2SO_4 soln. until the carmine-red colour disappears. (*ii*) In the amperometric

method, a rotating platinum micro-electrode is used during the titration of the SO_4'' with $Pb(NO_3)_2$. To 2 ml of aluminate liquor add conc. HNO_3 until the ppt. dissolves. Dilute to 20 ml, add 15 ml of ethanol and titrate with 0·1 *N* $Pb(NO_3)_2$. (*iii*) A sedimentation method is also described.

CHEM. ABSTR.

2971. **Determination of available sulphur in pyrites and pyritic concentrates from coal.** A. R. Panicker and N. G. Banerjee (*J. Sci. Ind. Res., B, India*, 1953, **12** [9], 417–420).—The sample is burnt in a current of air, and the mixed volatile products are passed successively through a quartz packing at 900° C and a sulphur-free carbon packing at 320° to 340°C. The amount of SO_2 absorbed in moist Cr_2O_3 is found by weighing. Duplicate determinations are completed in 1 hr. The results agree (± 0·15 per cent.) with those found by standard wet methods. S.C.I. ABSTR.

2972. **Rapid photometric determination of chromium in alloy steels and bronzes.** M. D. Kahn and F. J. Moyer (*Anal. Chem.*, 1954, **26** [8], 1371–1373).—Chromium is determined photometrically, as $Cr^{\cdots}$, in $HClO_4$ - H_3PO_4 - H_2SO_4 - H_2O_2 soln. at 585 mμ. The sample containing 10 to 150 mg of Cr is dissolved in acids [HNO_3 (10 ml) + HCl (10 ml) for alloy steels and dil. HNO_3 (1 + 1) (10 ml) for bronzes]. Seventy per cent. $HClO_4$ is added and the soln. is boiled until all the Cr is present as Cr^{VI}, and the soln. is cooled slightly and filtered. To the filtrate are added H_2SO_4 (10 ml) and 15 to 30 g of Zn, more being added during the reduction if necessary. After approx. 15 min., the mixture is filtered, and to the soln. is added 30 per cent. H_2O_2 (4 ml). To the boiled soln. is added H_3PO_4 (4 ml) and the mixture is filtered, the filtrate being made up to 200 ml. The light absorption is measured at 585 mμ and the chromium concn. is determined from standards. Speed of operation and precision (max. mean deviation is given as 0·37 per cent. in a series of 16 determinations) are stated to be good. C, Mn^{II}, P, S, V, Ti, Sn, Pb^{II}, Zn, Al, Cd, Nb^{V}, Ta^{V}, Se, Cu, W, Ni^{II}, Cu^{II}, Fe^{III}, Mo and Co^{II} do not interfere unless in high concn.

D. A. PANTONY

2973. **Methods for the analysis of iron and steel. XIII. Chromium in iron and steel.** British Standards Institution (B.S. 1121: Part 13: 1954, 8 pp.).—A solution of the sample in H_3PO_4 plus H_2SO_4 is oxidised with ammonium persulphate in the presence of $AgNO_3$. The formation of pink $HMnO_4$ is an indication of completeness of oxidation. For samples with Mn content < 1 per cent. a few drops of $KMnO_4$ solution are added. The $HMnO_4$ is reduced by boiling with HCl and the oxidised Cr is determined by adding an excess of ferrous ammonium sulphate and back-titrating with $KMnO_4$. The procedure is recommended as a referee method for a wide variety of plain and alloy irons and steels containing up to 25 per cent. of Cr. Reproducibility varies from ± 0·01 per cent. with 0·3 per cent. of Cr to ± 0·10 per cent. with 25 per cent. of Cr present. A modified procedure is recommended for steels containing over 2 per cent. of W.

C. J. KEATTCH

2974. **Polarographic determination of [sexavalent] molybdenum. Tartaric acid as supporting electrolyte.** E. P. Parry and M. G. Yakubik (*Anal. Chem.*, 1954, **26** [8], 1294–1297).—A soln. (< 30 ml), 1·5 × 10^{-4} to 4 × 10^{-3} *M* with respect to Mo, is treated with *M* tartaric acid (5 ml) and the pH is adjusted to 1·5 to 2·3; if $Cr^{\cdots}$ or $Fe^{\cdots}$ are present, SO_2 is passed into the hot soln. for 10 min. and the excess is removed by boiling. The cooled soln. is made up to 50 ml, and the polarogram of an aliquot under N is recorded. To the soln. in the polarograph cell is added a known vol. of standard Mo^{VI} (preferably sufficient to approximately double the Mo concn.) and a new polarogram is recorded. From the data and a given formula, the Mo concn. of the original soln. is calculated. V^{V} interferes seriously, but Cu^{II} can be tolerated up to a 10-fold excess. W^{VI}, Ni^{II}, Co^{II}, Fe^{III} and Cr^{III} do not interfere under the conditions described. D. A. PANTONY

2975. **Thiocyanate spectrophotometric determination of molybdenum and tungsten.** C. E. Crouthamel and C. E. Johnson (*Anal. Chem.*, 1954, **26** [8], 1284–1291).—Methods are described for the absorptiometric determination of Mo and W as their thiocyanato complexes, particularly those in acetone - water systems. The sample of Mo is dissolved in sufficient H_2SO_4 or HCl so that the final acid concn. is 0·3 to 1·0 *M* or ≯ 4 *M*, respectively. To the soln. are added 0·1 *M* Cu_2Cl_2 in conc. HCl (5 drops), followed by *M* NH_4CNS in acetone (15 ml) and then 10 per cent. $SnCl_2$ (5 drops). The soln. is made up to 25 ml with water and the light absorption is measured at 460 mμ and compared with standards. Alternatively, to the Mo^{VI} soln. in 8 *M* HCl is added 0·1 *M* Cu_2Cl_2 soln. (1 ml) (max. vol. of soln. is 3 ml), and the thiocyanate colour is developed with 3 *M* aq. NH_4CNS (3 ml). The complex is extracted with two 10-ml portions of ether and the extract is diluted to 25 ml with ether. Absorptions are measured at 470 mμ as soon as possible after extraction. W is determined similarly; the soln. (> 8 *M* in Cl' and > 10 *M* in $H^{\cdot}$) is reduced either with 10 per cent. $SnCl_2$ (2 ml) or with Hg (0·1 ml) and HCl (2 ml). Either (*a*) *M* NH_4CNS in acetone (15 ml) is added, and, after dilution to 25 ml, the absorption is measured directly at 398 mμ, or (*b*) 3 *M* aq. NH_4CNS (5 ml) is added, and, after extraction with two 10-ml portions of ether and making up to 25 ml with ether, the absorption of the extract is measured at 405 mμ. Alternatively the extraction in (*b*) can be omitted and the absorption can be measured directly at 398 mμ. Factors influencing colour developments and avoidance of interference from $Fe^{\cdots}$ are discussed. D. A. PANTONY

2976. **Potentiometric determination of tellurium.** A. M. Amin, I. M. Issa and R. M. Issa (*Chemist Analyst*, 1954, **43** [3], 61–63).—Ten ml of an aq. solution of tellurite (3 × 10^{-5} *N*) are placed in a 25-ml beaker fitted with a rubber bung and provided with holes for a micro-burette, a mechanical stirrer, a salt bridge and a platinum electrode 1·5 mm in diameter. The salt bridge contains saturated KCl and is connected to a calomel electrode. The solution is titrated in 0·05-ml increments with 10^{-4} *N* $KMnO_4$ solution and the end-point is observed potentiometrically. The platinum electrode must be etched with aqua regia before each titration. With quantities of Te as low as 1 μg in 10 ml, the error is 1 to 2 per cent. H. P. PAGET

2977. **Polarographic determination of tungsten in rocks.** L. E. Reichen (*Anal. Chem.*, 1954, **26** [8], 1302–1304).—A method, claimed to be rapid and accurate, for the determination of W in rocks by polarography in 4·6 *M* HCl - 0·1 *M* tartrate soln. is described. The rock sample (0·5 g) is fused with Na_2CO_3 (5 g) and the cooled melt is leached with water (20 ml). After filtration, the soln. is made up to 50 ml; a 10-ml aliquot of this is treated with *M* aq. sodium tartrate (5 ml), thymol blue soln. (0·1 ml),

sufficient conc. HCl to produce a pink colour and then 20 ml in excess, 5 per cent. alcoholic cinnamic acid (0·25 ml) and water to 50 ml. On a portion of this mixture a polarogram is taken at −0·78 V (corresponding to the second and most distinctive $E_{\frac{1}{4}}$), and the tungsten concn. is derived by reference to standards prepared similarly. Mean deviation is ≃ 0·57 mg. There is no interference by CO_3'', V, Fe$^{\cdot\cdot}$ and small concn. of Mo, but HSO_4' (unless neutralised with Na_2CO_3) and NO_3' interfere seriously. D. A. PANTONY

2978. **Specific atomic grouping in reagents for tungstates.** I. M. Korenman, and E. I. Levina (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 170–174).—The organic reagents for detecting tungstates contain the grouping ·CH(OH)·CHX· or ·CH(OH)·CH_2·CHX·, where X is an atom of O or N forming a co-ordination bond, and the C atoms can be present in aliphatic or in cyclic compounds. The reaction with tungstates depends on the formation of five- or six-membered heterocyclic internal-complex esters of tungstic acid. G. S. SMITH

2979. **Uranium determination by use of the photo-decomposition of oxalic acid.** H. H. Paige, A. E. Taylor and R. B. Schneider (*Science*, 1954, **120**, 347–348).—Shallow Pyrex-glass dishes containing samples are placed in an enclosed space approx. 4 in. from a battery of six 15 W Sylvania or G.E. germicidal lamps giving 90 per cent. radiant energy at the 2537 A line. The area of the light source is approx. 324 sq. in. and the area used for exposure of samples approx. 34 sq. in. Five standards were prepared in 500-ml calibrated flasks as follows. Ten ml of 0·1 *M* oxalic acid were placed in each flask followed by 70, 50, 30, 10 or 0 ml of standard uranyl nitrate soln. (0·0117 mg of U per ml). Each flask was filled to the mark with demineralised H_2O and thoroughly mixed. Eight 15-ml aliquots of each were exposed to u.v. light for 2 hr. and rinsed into titration flasks, 5 to 10 ml of 18 *N* H_2SO_4 were added and the solutions were diluted to 100 ml with H_2O and titrated with 0·002 *N* $KMnO_4$. The vol. of $KMnO_4$ used was plotted against μg of U per litre to provide a standard graph that was used to determine U in another series of samples prepared and treated as above. In the range 10^{-4} g per litre, the standard deviation was less than 10^{-5} g per litre; many variables remain to be studied. H. F. W. KIRKPATRICK

2980. **Estimation of impurities in liquid chlorine by infra-red absorption spectrophotometry.** A. W. Pross (*Nature*, 1954, **174**, 467).—A 5·25-cm tantalum-clad steel pressure cell with calcium fluoride windows is filled with liquid Cl, and spectra are recorded in the range 4000 to 1180 cm^{-1}, in which pure liquid Cl is completely transparent. Concentration of absorbing impurities are determined from application of Beer's law at max. absorption by use of known additions of chloroform, methylene chloride and hexachlorobenzene. Five p.p.m. of these impurities can be detected with an accuracy of ± 10 per cent. at 50 p.p.m. Moisture may be estimated by the use of the band at 1640 cm^{-1}; CO_2 does not interfere. H. P. PAGET

2981. **The micro-determination of bromide in presence of chloride.** G. Hunter and A. A. Goldspink (*Analyst*, 1954, **79**, 467–474).—The method described depends upon quant. oxidation of Br′ to BrO_3' by OCl′ in a suitably buffered soln., reaction of BrO_3' with excess of Br′ to liberate free Br and combination of the liberated Br with decolorised rosaniline, the tetrabromorosaniline being then determined absorptiometrically at 570 mμ. The method is independent of the presence of Cl′, and I′ interferes only when it is greatly in excess of Br′. Interference by ClO_3' (formed by auto-oxidation of HOCl) is prevented by addition of ammonium molybdate. This catalyses the reaction between Br′ and BrO_3' at a degree of acidity lower than that necessary to cause significant reaction between ClO_3' and Br′. The absorptiometric procedure is applicable to amounts of Br up to 5 μg in 5 ml of soln. In this vol., 0·1 μg of Br′ can be measured accurately and even smaller amounts measured by reducing the vol. A. O. JONES

2982. **Micro-volumetric determination of manganese.** H. Flaschka, A. M. Amin and R. Zaki (*Chemist Analyst*, 1954, **43** [3], 67–69).—Mn can be determined by titration with ethylenediaminetetra-acetic acid (**I**), provided that Fe and Al are previously removed with ZnO. To a solution containing 300 to 500 μg of Mn, ZnO is added in 0·1-g increments until precipitation is complete; the solution is filtered through a porcelain filter-stick and washed with 1 ml of H_2O. To the filtrate are added 0·1 g of KCN, 0·1 g of Rochelle salt and 0·1 g of ascorbic acid, followed by 2 ml of buffer solution (54 g of NH_4Cl and 352 ml of conc. aq. NH_3 made up to 1 litre) and Eriochrome black T indicator. The solution is warmed to 70° to 80° C and titrated with a 0·01 *M* solution of **I**. It is shown that Mn is not adsorbed by the ZnO in the above procedure and that PO_4''' does not interfere. H. P. PAGET

2983. **Flame-spectrum analytical investigation of heavy metals for determining the ferrite composition of steel.** F. Wever, W. Koch and G. Wiethoff (*Arch. Eisenhüttenwesen*, 1953, **24**, 383–391).—The advantages of flame-spectrum analysis for the quick determination of the composition of steels are pointed out, and the principles of application are described with particular reference to the composition of the ferrite dissolved in the citrate electrolyte in the electrolytic isolation of the carbides in Cr - Mn steels. An arrangement of especially high measuring sensitivity was developed which permits the determination of Fe, Cr and Mn in presence of considerable excess of alkali salts originating from the electrolyte. The influence of the composition of the electrolyte and that of the combustion gases of the flame on the intensity of the flame spectrum is discussed, and an example for a complete determination of the distribution of Cr and Mn in carbide and ferrite is given. CHEM. ABSTR.

2984. **The determination of some heavy metals (present together) by a chromatographic method.** O. I. Khokhlova (*Aptechnoe Delo*, 1954, **3** [3], 17–23).—The results of experiments on the separation of heavy metal ions on alumina columns and on filter-paper strips are given. Co^{II} is separated from Ni and Fe, NH_4CNS being used as developer; the Ni^{II} zone is made visible by means of dimethylglyoxime. When saturated KI soln. is used as a developer, Hg^{II} can be separated from Fe^{III}, Cr^{III} from Fe^{III}, and Cu^{II} from Pb^{II}. The methods can be applied to the detection and determination of the metals when only small amounts are present. E. HAYES

2985. **Ultra-violet spectrophotometric titrations with disodium Versenate [ethylenediaminetetra-acetate].** D. C. Burtner (*Dissert. Abstr.*, 1954, **14** [5], 755–756).—Procedures are described for the volumetric analysis of Fe, Cu, Ni and Co chelates of disodium ethylenediaminetetra-acetate by a high-absorbance reference method. An accuracy of 0·4

per cent. is claimed for metal ion concn. $\gtrsim 0{\cdot}01\ M$, and solutions as dilute as $10^{-4}\ M$ can be titrated.
L. F. TAYLOR

2986. **Methods for the analysis of iron and steel. XXX. Cobalt in iron and steel. (Absorptiometric method.)** British Standards Institution (B.S. 1121: Part 30: 1954, 7 pp.).—The sample is dissolved in H_3PO_4 plus H_2SO_4, HNO_3 is added and the solution is evaporated to fumes. After cooling, the solution is transferred to a 100-ml calibrated flask and made up to volume with water. To an aliquot is added nitroso-R-salt followed by Na acetate solution. After mixing and setting aside for 5 min., HNO_3 is added and the solution is let stand for a further 5 min.; it is then diluted to 100 ml and the absorption is measured at 560 to 600 mμ. A control test is carried out, no nitroso-R-salt being added. The percentage of Co is derived from a previously prepared graph. The procedure has been tested on a variety of alloy steels containing up to 12 per cent. of Co, and the reproducibility is ± 0·05 per cent. at the 2 per cent. cobalt level. It is recommended as a referee method. Up to 5 per cent. of Ni does not interfere and, in the absence of Ni, up to 2 per cent. of Cu can be tolerated. Higher percentages of these elements require the addition of more nitroso-R-salt.
C. J. KEATTCH

2987. **Mercaptoacetic [thioglycollic] acid as a complexing reagent for the determination of nickel.** E. Trepka-Bloch (*Chemist Analyst*, 1954, **43** [3], 63–65).—The method described is successful in presence of Fe, Co, Cu, Ag, Pb, As and Bi. Cobalt concentrate (0·2 g) is decomposed with 10 ml of aq. HNO_3 (1 + 1). When most of the sample is decomposed, HF, a little $KClO_3$ and, finally, 10 ml of 70 per cent. $HClO_4$ are added. After heating to fuming and adding 5 ml of conc. HCl, the solution is heated to dissolve salts and the Fe is reduced with Na_2SO_3. After filtering off silica, 6 ml of thioglycollic acid (95 per cent.) are added with 5 g of Na_2SO_3, 330 ml of H_2O, and 20 ml of conc. aq. NH_3; the solution is heated to 60° C, and 50 ml of 3·33 per cent. w/v Na dimethylglyoxime soln. are added. The mixture is left for 20 min. at 45° C. After filtration, the ppt. is washed with hot water and then extracted with 100 ml of hot aq. hydrochloric acid (1 + 2). Organic matter in the filtrate is destroyed by boiling with 1 g of $KClO_3$; Na citrate (1 g) and 0·1 per cent. $AgNO_3$ soln. (5 ml) are added, and the solution is diluted to 150 ml and made weakly alkaline with aq. NH_3. Five ml of 4 per cent. w/v KI soln. are added, and the solution is titrated with KCN soln. (8·6 g of KCN and 5 g of KOH per litre) the titre corresponding to the $AgNO_3$ alone being deducted.
H. P. PAGET

2988. **Spectrophotometric studies of reactions of importance in colorimetry. Complex compounds formed by nickel ions with the sodium salt of 2-nitroso-1-naphthol-4-sulphonic acid.** V. N. Tolmachev and L. A. Korobka (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 134–140).—In solutions containing Ni^{++} and 2-nitroso-1-naphthol-4-sulphonate (R′) two complexes NiR^{+} and NiR_3' can form, according to the pH. The instability constants are $1{\cdot}3 \times 10^{-6}$ and $1{\cdot}5 \times 10^{-14}$, respectively. NiR_3' is fairly stable at pH values as high as 10. It has a high molar extinction coeff. and may be of interest for the colorimetric and spectrophotometric determination of Ni.
G. S. SMITH

2989. **Spectrophotometric study of *p*-nitrosodimethylaniline as a sensitive colorimetric reagent for platinum.** J. J. Kirkland and J. H. Yoe (*Anal. Chem.*, 1954, **26** [8], 1340–1344).—The factors influencing the production of the orange-red colour produced by $PtCl_6''$ and dimethyl-*p*-nitroso-aniline reagent (temp. of mixing, time of heating, concn., choice of solvent, pH and interferences) are examined, and the light absorption characteristics of the complex are investigated. The results are used for the simultaneous determination of Pt and Pd described in Abstract 2990 below. The Beer-Lambert law holds up to concn. of 3·2 p.p.m. of Pt, and the limit of sensitivity is given as 0·015 p.p.m. in a 1-cm spectrophotometer cell. Rh, Ir, Os and Ru all give colours with the reagent and interfere, as do high concn. of $AuCl_4'$, Fe^{+++}, Cu^{++}, Ni^{++}, Co^{++} and Cr^{+++}.
D. A. PANTONY

2990. **Separation of platinum and palladium and their subsequent colorimetric determination with *p*-nitrosodimethylaniline.** J. H. Yoe and J. J. Kirkland (*Anal. Chem.*, 1954, **26** [8], 1335–1339).—Dimethyl-*p*-nitroso-aniline is employed as a colorimetric reagent (see Abstract 2989 above) for determination of Pt and Pd together by three possible methods. (*a*) The soln. containing 12 to 40 μg of Pd and 35 to 120 μg of Pt is adjusted to pH 2 to 3, and the vol. made up to 10 ml with water. To this is added sodium acetate - HCl buffer (50 ml of sodium acetate and 53 ml of 4 *M* HCl) (1 ml) and dimethyl-*p*-nitroso-aniline (5 mg per ml of ethanol) (1 ml), and the soln. is either heated at 100° C for 20 min. after addition of water (5 ml) or heated at 85° C for 60 min. after addition of ethanol (4 ml). After cooling and dilution to 50 ml with 95 per cent. ethanol, the total Pt and Pd concn. is determined spectrophotometrically at 525 mμ; Pd only is determined by a similar procedure on another aliquot for which the heating is omitted. Pt is obtained from the difference of absorption between the two methods. Comparison with standards treated similarly gives the Pt and Pd concn. separately. (*b*) At pH 2 to 5 the platinum - palladium soln. (≃ 10 ml) containing $\gtrsim 500$ μg of Pd is treated with dimethyl-*p*-nitroso-aniline reagent (0·5 ml) and 95 per cent. ethanol (5 ml) in a separating funnel. The red colour due to Pd is extracted with $CHCl_3$ and the operation is repeated. The organic and aq. phases are evaporated separately and the organic matter is destroyed by baking and treatment with HNO_3 and H_2O_2. NO_3' is removed by successive fuming with HCl. The determinations are carried out on the chloride solutions as described in (*a*): hot treatment for platinum and cold for palladium determinations. (*c*) The diethyldithiocarbamate complexes are prepared and extracted into benzene by standard methods; after removal of organic matter, the method described in (*a*) is applied for the determination of Pd and Pt. Errors for each method are given as ± 1·5 per cent. for Pt and ± 1 per cent. for Pd.
D. A. PANTONY

2991. **Chromatographic semi-microanalysis of gases. I. Theoretical and practical basis of analysis.** J. Janák (*Chem. Listy*, 1953, **47** [6], 817–827).—The method enables the rapid (5 to 30 min.) and quant. determination of individual components of a gas mixture (1 to 30-ml samples) by measurement of their volumes eluted in certain sequence from activated charcoal or silica gel by CO_2 as eluting gas. A discussion of theory is followed by a detailed description of the apparatus, which consists in a horizontal adsorption column, a micro-burette for measuring the vol. of the sample, a nitrometer filled with conc. KOH, and devices for measuring and regulating the flow of the purified

and dried eluting gas. The influence of the rate of flow of the eluting gas, of the size and shape of the adsorption column, of the vol. of sample, of temp., and of the grain size of the adsorbent is discussed. The method is limited to non-acidic gases, to gases insol. in, or not reacting with, conc. KOH, and to gases chemically unaffected by the catalytic action of the adsorbent. G. GLASER

2992. **Chromatographic semi-microanalysis of gases. II. Analysis of natural gas and determination of methane in mine gases.** J. Janák (*Chem. Listy*, 1953, **47** [6], 828–836).—The characteristic elution volumes (chromatographic spectra) of the components of natural gas on activated charcoal have been determined. The R_F values of the gases have then been calculated from $R_F = V/V_{max.}$, where V is volume of eluting gas in the gas phase of the column, and $V_{max.}$ is the characteristic elution volume. R_F values at 20° C are: H, 0·6429; N and O, 0·3025; methane, 0·1169; ethane, 0·0059. At 85° C R_F values are: N, 0·7347; methane, 0·5143; ethane, 0·0935; propane, 0·0160; *n*-butane, 0·0117. Optimum conditions for the chromatographic separation of a given mixture have been calculated, and practical examples of analyses of natural gas and of mine atmospheres are given. The influence of the temp. of the adsorbent on the reproducibility and accuracy of the method is discussed; adsorption temp. > 90° C cause reduction of CO_2 to CO. One filling of the column lasts for several hundreds of analyses, but for only 50 to 70 if H_2S is present. G. GLASER

2993. **Chromatographic semi-microanalysis of gases. III. Analysis of gases rich in hydrogen.** J. Janák (*Chem. Listy*, 1953, **47** [6], 837–841).—A number of industrial gases containing H (1 to 3-ml samples) were analysed at 20° C on activated charcoal during 15 to 30 min. These included water gas, town gas, synthesis gas, carbonisation gas, a mixture of methane and ethane, and crude H. From the characteristic elution volumes of individual components the following R_F values were derived: H, 0·643; N, 0·3025; O, 0·307; CO, 0·244; methane, 0·117. It follows that the separation of H from methane is easy; N and O cannot be separated. Overlapping of the nitrogen and carbon monoxide zones can be overcome by using a column with a large volume of adsorbent. Irreversible sorption, noticeable in such cases, can be prevented by flushing the column with a 3-ml sample of the gas to be analysed before analysis. G. GLASER

2994. **Analysis of gases [and particularly the determination of traces of oxygen].** Mond Nickel Co., Ltd. (Inventor: P. Hersch) (Brit. Pat. 707,323. Date Appl. 16.1.52).—The gas is fed to a galvanic cell in which the O is absorbed at the cathode (a metal such as Ag that is not attacked by the electrolyte in the presence or absence of O) to produce an electric current that is a function of the O concn. The anode (*e.g.*, saturated antimony amalgam or porous Ni saturated with Cd) is readily attacked by the electrolyte (*e.g.*, aq. KOH) in presence of O, but not in absence of O.
J. M. JACOBS

2995. **Accurate analysis of waste gas with a soap-film apparatus.** W. J. Gooderham (*Chem. & Ind.*, 1954, [5], 121–122).—A soap film method similar in accuracy to the Haldane gas analysis apparatus has been devised. The apparatus contains four soap film meters (*Brit. Abstr. C*, 1953, 413) calibrated to allow estimation to 0·01 per cent., and apart from a blower to deal with gases at or below atmospheric pressure is similar to that previously described (*Brit. Abstr. C*, 1949, 320). The following absorbents were used, soda lime for carbon dioxide, ferric sulphide followed by soda lime for oxygen, and a Katz - Halpern reagent followed by soda lime for carbon monoxide. The Katz - Halpern reagent was prepared by mixing α- Fe_2O_3 (70 g) and a solution of $AgNO_3$ (80 g in 110 g of water) to form a paste that was then added to a solution of $KMnO_4$ (83 g in 1200 g of water) and cooled to 5° to 6° C. After 1 hr. the sludge was filtered; the residual paste was dried at 60° C in a stream of air for 1 hr., and made into pellets. D. LIFF

2996. **Quantitative radiochemical method for determination of major sources of natural radioactivity in ores and minerals.** J. N. Rosholt, jun., (*Anal. Chem.*, 1954, **26** [8], 1307–1311).—A method for determining the degree of equilibrium of radioactive series in ores is described. The sample (0·1 to 0·5 g) is fused strongly with Na_2O_2 (4 g) under standard conditions, and the cooled melt is leached with water, neutralised with HCl, and its concn. finally adjusted to N in a total vol. of 100 ml. $Bi^{\cdots}$ (1·34 g of Bi_2O_3 dissolved in 5 ml of HCl, made up to 200 ml) and $Pb^{\cdot\cdot}$ (0·48 per cent. $Pb(NO_3)_2$ soln.) (1·0 ml each) are added and H_2S is passed in at 45° to 50° C for 10 min. The ppt. is filtered on paper, washed with water and finally ethanol, and then carefully dried. Times of operations are recorded. The ppt. may contain sulphides of polonium-218, -214, -212 and -210 and bismuth-214, -212 and -210, the radioactivity of which are selectively determined by α-counting on a scintillation detector, as follows. *Radon group*—Controlling isotope is ^{214}Bi. Count is taken on the 7·68 MeV α-particles of ^{214}Po as soon as possible after pptn. *Radium-228 group* (^{232}Th)—Controlling isotope is ^{212}Bi after decay of ^{214}Bi. Count is taken of the 8·78 MeV α-particles of ^{212}Po. *Radon-222 plus Thorium-232*—Count is taken of the combined ^{212}Bi and ^{214}Bi, and after the decay of ^{214}Bi, the emission of ^{212}Bi alone is counted. *Lead-210 group*—One day or more after pptn., the α-count of ^{210}Po is taken and compared with that of a standard source of ^{210}Po. *Radium-226 group*—The α-count is taken of volatilised ^{222}Rn and compared with that of a standard sample. Extrapolation of the plots of time against log[α-count] gives a measure of the concn. of the parent element of each group series. The ^{238}U series is examined by standard chemical methods. Errors are given as 5 to 8 per cent. Limitations (*e.g.*, solubility of the ores in Na_2O_2, etc., and high concn. of heavy metals that lead to bulky sulphide precipitates with consequent self-absorption of α-particles) and sensitivity are discussed. D. A. PANTONY

2997. **Recent trends in glass analysis.** F. W. Glaze (*Bull. Amer. Ceram. Soc.*, 1954, **33** [2], 45–48).—The rapid methods used in cement and rock analysis are described. Methods for the determination of SiO_2, B_2O_3, total iron, Fe^{II}, Zn, CaO, MgO, soda, potash, F and Cl are reviewed. The published procedures for determining small amounts of colorants such as Cr, Co, Ni and Cu are also mentioned. B. J. W.

2998. **Operations involved in cement analysis.** L. Bean and E. J. Hackney (*Bull. A.S.T.M.*, 1954, No. 197, 43–48).—From an examination of data (listed) for 0·5-g samples, answers are given to the usual questions arising from the use of various

procedures in different laboratories for the analysis of cements. The SiO_2 ppt. should be ignited for 1 hr. at 1200° C, and the R_2O_3 ppt. for 1 hr. at 1100° C; in both instances loss of Pt from pure platinum crucibles is negligible, whilst there is no appreciable reduction of Fe_2O_3 to Fe_3O_4 in the mixed oxides. Precipitates should be ignited without flaming of the filter-paper. The HF residue should be ignited at 1050° to 1100° C for 5 min. to ensure constant wt. Results are slightly higher with platinum evaporating dishes than with porcelain dishes or Pyrex-glass beakers when used for the double dehydration of SiO_2. One pptn. of Ca oxalate in a vol. of $\simeq$ 200 ml only, followed by titration with $KMnO_4$, gives satisfactory values for CaO. The kind of filter-paper used and the vol. of wash-water are not critical, but greater accuracy is attained by adding ammonium oxalate to an acid soln. at 70° to 80° C and then neutralising with aq. NH_3 against methyl red indicator. After keeping for 1 hr., the soln. is filtered and the ppt. is washed with 75 ml of hot water before preparation of the titrating soln. W. J. BAKER

See also Abstracts 2915, 3101.

3.—ORGANIC ANALYSIS

2999. **Rapid methods of microanalysis. Simultaneous determinations of carbon, hydrogen and silicon by combustion in the presence of catalysts.** V. A. Klimova, M. O. Korshun and E. G. Bereznitskaya (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **96** [1], 81-83).—Combustion in the presence of V_2O_5 or CrO_3 supported on asbestos prevents the formation of SiC in the combustion of organo-silicon compounds, and yields quantitative figures for silicon content. R. C. MURRAY

3000. **Detection and determination of the carbonyl group by spectrophotometry in the ultra-violet.** P. Rumpf (*Chim. Anal.*, 1954, **36** [9], 229–233).—The methods and theory of u.v. spectrophotometry are briefly reviewed. The effect of different solvents and structural environment of the group on the spectrum of the carbonyl group is discussed in detail, and the spectra of diketones and of 2:4-dinitrophenylhydrazones are also considered. E. J. H. BIRCH

3001. **Proteins. XXI. Determination of thiol groups by polarographic titration.** L. Matoušek and O. Laučíková (*Chem. Listy*, 1953, **47** [7], 1062–1065).—Free thiol groups in proteins are specifically determined by polarographic titration (dropping-mercury electrode) with 0·001 to 0·0001 *M* Na *p*-chloromercuribenzoate. The method, which is accurate to $\pm$ 3 per cent., has been tested on 10 to 30-mg quantities of cysteine, albumins and globulins. Cystine and methionine do not interfere. G. GLASER

3002. **Micro-determination of arsenic in organic compounds. Application of potentiometric argentimetry by reference potentials.** R. Lévy (*Compt. Rend.*, 1954, **238** [24], 2320–2321).—The procedure, which involves pptn. of Ag_3AsO_4 from soln. (pH 8·8) with $AgNO_3$, followed by back-titration of the excess of $AgNO_3$ with KCl, is the same as that described previously for the micro-determination of phosphorus (*Brit. Abstr. C*, 1953, 380) except that the $AgNO_3$ and KCl solutions used in the potentiometric titrations are 0·015 *N*, and that no further addition of $AgNO_3$ soln. is necessary after the reactants are diluted with H_2O (equilibrium being reached instantaneously). The error (ϵ) in the arsenate titre (V') is given by $\epsilon = 0{\cdot}223 \log_{10}(1 + V') + 0{\cdot}058$ for a final vol. (before filtration) of 80 ml at 20° C. If V_1 is the total vol. of $AgNO_3$ added, v_1 the back-titre (with KCl) of excess $AgNO_3$, then $V' = (V_1 - v_1)$ and V_0 (true arsenate titre) = $(V' - \epsilon)$. W. J. BAKER

3003. **Determination of tetraethyl-lead.** J. C. Chaudhuri (*J. Indian Chem. Soc., Ind. News Ed.*, 1953, **16** [4], 207–216).—A review of published methods for determination of $Pb(C_2H_5)_4$, with 32 references. M. TADMAN

3004. **Determination of olefin group types. Chromatographic and infra-red absorption techniques.** E. L. Saier, A. Pozefsky and N. D. Coggeshall (*Anal. Chem.*, 1954, **26** [8], 1258–1263).—Group type analyses, by means of i.r. absorption techniques, for the olefinic functional groups RCH:CH·, RR'C:CH·, RCH:CHR'· (*cis* and *trans*), and RR'C:CR''· are described. Chromatographic separations were used as a means of concentrating the olefins and minimising interferences by non-olefinic compounds. A high degree of accuracy is not possible owing to the variation in absorption among olefins of the same class. The utilisation of dil. CS_2 solutions of the olefins with a comparatively thick cell is also described. G. P. COOK

3005. **Studies with N-halo reagents.** P. F. Kruse, jun., K. L. Grist and T. A. McCoy (*Anal. Chem.*, 1954, **26** [8], 1319–1321).—N-Bromo, N-chloro and N-iodosuccinimides were used as reagents for the differentiation of primary, secondary and tertiary alcohols and amines, and some position isomers, and for the detection of amino-alcohols. Thirty saturated alcohols, 25 other alcohols and hydroxy compounds were tested with N-bromosuccinimide; 30 saturated amines were tested with N-iodosuccinimide and 22 position isomers were detected by various N-halosuccinimides. In general primary alcohols with the bromo reagent give a permanent orange colour, secondary alcohols give an orange ppt. and tertiary alcohols give no colour change. With the iodo reagent, primary amines give a permanent brown colour, whereas the colour fades with secondary amines. Tertiary amines give a brown colour and are confirmed by an orange ppt. with N-bromosuccinimide. The tests were carried out in CCl_4 soln. at 80° C. G. P. COOK

3006. **Rapid determination of *n*-butanol, acetone and ethanol in industrial butyl fermentations.** J. Sisquella Argila (*Inf. Quím. Anal.*, 1954, **8** [4], 133–138).—The fermentation mixture is neutralised and a third of it is distilled. Acetone is determined iodimetrically on a portion of the distillate. The alcohols are determined by carrying out a quant. oxidation with $K_2Cr_2O_7$ on a further portion of the distillate, and then a second oxidation either after extraction of a portion of the *n*-butanol with CCl_4 or under strongly acid conditions. These two oxidations give simultaneous equations from which, with a known concn. of acetone, the concn. of *n*-butanol and ethanol can be calculated. L. A. O'NEILL

3007. **Colour reactions of aldehydes.** L. Rosenthaler and G. Vegezzi (*Mitt. Lebensm. Hyg., Bern.*, 1954, **45** [3], 178–182).—The behaviour of 43 aldehydes with the following reagents is reported: (*a*) fuchsin - sulphurous acid, (*b*) diazobenzenesulphonic acid, (*c*) phenylhydrazine followed by diazobenzenesulphonic acid and (*d*) *m*-phenylenediamine. With (*a*), salicylaldehyde, *p*-hydroxybenzaldehyde, vanillin, "ethylvanillin," 2:4-dihydroxybenzaldehyde and chloral hydrate give no

coloration; glyoxal and *m*-hydroxybenzaldehyde give a positive reaction. Aldehydes that fail to give a positive reaction with (*b*) even after the addition of sodium amalgam include: α-phenylpropionaldehyde, *m*- and *p*-hydroxybenzaldehydes, *p*-dimethylaminobenzaldehyde, *m*- and *p*-nitrobenzaldehydes and the higher aliphatic aldehydes. With (*c*) and (*d*), negative results are less common, but none of the reagents gave positive results with all the aldehydes tested. E. HAYES

3008. **Volumetric determination of aldehydes and ketones with phenylhydrazine hydrochloride.** A. P. Terent'ev and K. S. Zabrodina (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **95**, 85–87).—Experimental procedure for the volumetric determination of aldehydes and ketones is described in detail. Excess of phenylhydrazine hydrochloride is added to the analysed solution, which after being left in darkness for several hours is titrated with a standard cupric acetate solution. The percentage and number of carbonyl groups are then estimated after taking into account the amount of cupric acetate used up in a blank test. Results of determinations of 14 aldehydes and ketones are reported, good agreement with theoretical values being found.
S. K. LACHOWICZ

3009. **Analysis for industry. [Determination of acetic acid.]** A. Sykes (*Ind. Chem.*, 1954, **30** 340–341).—Chemical and instrumental methods are described for determining acetic acid in the presence of other organic materials and in biological samples, and for assessment of quality. (27 references.)
L. F. TAYLOR

3010. **Analysis for industry [Determination of lower fatty acids.]** A. Sykes (*Ind. Chem.*, 1954, **30**, 283–285).—Recent methods for analysing mixtures of formic, acetic, propionic and butyric acids, particularly those based on distillation and partition between H_2O and immiscible organic solvents, are reviewed. (33 references.)
D. R. PECK

3011. **Paper chromatography of fatty acids.** P. Savary (*Bull. Soc. Chim. Biol.*, 1954, **36** [6–7], 927–932).—The chromatographic separation of stearic, palmitic, myristic, lauric, capric, oleic and linoleic acids on 3 grades of ordinary or silicone-treated filter-paper is described. With untreated filter-paper, the solvent system used is 50 to 90 per cent. aq. methanol, and the positions of the acids are revealed by the following procedures. The papers are soaked in 1 per cent. cupric acetate; Whatman paper is then washed and treated with 0·1 per cent. $K_4Fe(CN)_6$, but Schleicher and Schüll papers retain Cu and are therefore dried at 60° C and treated with 10^{-4} *N* $KMnO_4$. Both types of paper are also treated with 0·1 per cent. Rhodamine B after cupric acetate, and observed under a Wood's lamp. Silicone-treated papers are prepared by dipping them for 10 sec. in a 1 per cent. solution of Rhodorsil 240 (Rhone-Poulenc) in anhydrous *cyclo*hexane and drying them vertically for 2 days. Mixtures of aq. acetone and *cyclo*hexane, *n*-hexane or petrol (boiling range 100° to 120° C) are used as solvents, and the acid positions are shown with cupric acetate and Rhodamine B, which is applied on the reverse of the paper only. The most suitable techniques for various acid mixtures are discussed. C. E. SEARLE

3012. **Paper chromatography of organic acids.** F. Smith and D. Spriestersbach (*Nature*, 1954, **174**, 466–467).—In paper chromatography uronic and hydroxy acids often show streaked spots; these spots can be prevented by depositing alginic acid on the paper by dipping it successively in ammonium alginate soln. and *N* HCl. Any developing solvent may then be used, and reproducible R_F values are attained. Paper so treated may be used for partition chromatography of D-mannuronic, D-glucuronic, D-galacturonic, citric, tartaric, succinic, malic, glycollic and malonic acids, and also various aldobiuronic acids. Acid - base indicators cannot be used for detection, but ammoniacal $AgNO_3$ and aromatic amine sprays are suitable; alternatively the paper may be sprayed with a 50 per cent. aq. alcoholic soln. containing 5 per cent. of an amine and 5 per cent. of $NaNO_2$, and subsequently with an alkaline solution of a phenol or an aromatic amine. This gives azo-dye spots readily detectable against the fluorescent background. In another method, the paper is sprayed with a solution containing 5 per cent. of aromatic amine and 5 per cent. of reducing sugar, and then heated for a short time at 125° to 130° C; brown spots indicate the presence of acids. H. P. PAGET

3013. **The volumetric determination of organic salts in mixtures of acetone and water solution.** Ts. I. Shakh (*Aptechnoe Delo*, 1954, **3** [2], 25–30).—Salts of organic acids can be titrated in aqueous acetone solution against 0·1 *N* HCl with methyl orange as indicator. The concn. of acetone in the soln. at the end-point should be 30 per cent. for Na salicylate and Ca gluconate, 25 per cent. for Na benzoate and Na citrate, and 20 per cent. for calcium lactate. Results obtained on these salts agree with those obtained by the standard (Soviet Pharmacopoeia VIII) methods. Sodium benzoate and sodium salicylate can be estimated in mixtures containing sucrose, phenacetin, K guaiacolsulphonate, acetylsalicylic acid or caffeine, but when phenazone is present the results are too high. E. HAYES

3014. **Absorption spectrum of aqueous monochloramine solutions.** J. Kleinberg, M. Tecotzky and L. F. Audrieth (*Anal. Chem.*, 1954, **26** [8], 1388–1389).—Monochloramine (**I**) gives an absorption max. at 243 mμ in solutions buffered over the pH range 9 to 11. The molar extinction values for this pH range are between 450 and 460, and with 1-cm cells the max. appears in solution containing as little as 5×10^{-4} *M* **I**. Beer's law is obeyed over the concn. range $64{\cdot}5 \times 10^{-5}$ to $310{\cdot}5 \times 10^{-5}$ *M*. The solutions are fairly stable, greatest decomposition occurring at the lower pH values. The possible decomposition products of aq. **I** do not absorb to any degree at 243 mμ. G. P. COOK

3015. **Use of αα′-diacetylenic ketones for the identification of primary and secondary amines.** J. Chauvelier (*Bull. Soc. Chim. France*, 1954, **21** [6], 734–739).—The reactions described previously (*Brit. Abstr. A II*, 1949, 130 and 468) are reviewed and extended to provide a method for identifying most primary or secondary amines available only in amounts of $\simeq$ 1 dg. On mixing equimol. amounts of dipropynyl ketone (**I**) and a secondary amine (both dissolved in the min. vol. of ethanol), there is formed at room temp., or on heating, a cryst. additive compound, the m.p. of which characterises the original amine. Primary amines yield similarly an additive compound, which, on heating slowly in xylene soln., yields the isomeric N-alkyl or N-aryl lutidone of specific m.p. With di-(2-phenylethynyl) ketone (**II**) instead of **I**, are formed, after several hours of heating of the additive compound in xylene, two crystalline isomers—the corresponding pyridone and an orange-coloured compound

—which generally form an admixture on cooling. These two isomers can be effectively separated, as the pyridone only is soluble in warm ethanol, whilst the orange-coloured isomer only is very sol. in warm benzene; each isomer can then be purified by reversing the solvents and its m.p. determined. The use of both **I** and **II** thus provides, in certain instances, five different specific m.p. for identifying a primary amine (except diphenylamine, *o*-nitroaniline, dichloroanilines, and long-chain amines, which do not react as above). The precautions necessary to ensure complete isomerisation, absence of resinous by-products and effective recrystallisation are described. Melting points and nitrogen contents of the additive compounds, lutidones, and orange-coloured isomers formed by reaction of **I** or **II** with a large number of primary and secondary aliphatic and aromatic amines are listed.

W. J. BAKER

3016. **Systematic method for identification of amines by colour reactions. II. Aromatic amines.** I. Bertetti (*Ann. Chim., Roma*, 1954, **44** [5–6], 313–320).—A systematic method for the identification of single aromatic amines consists in their successive treatment with the following 5 reagents: 5 per cent. aq. $K_2Cr_2O_7$ - conc. H_2SO_4 (2 + 1); 10 per cent. aq. $FeCl_3$; 10 per cent. aq. KNO_2; 8 per cent. aq. furfuraldehyde; and dil. aq. Br. The colours or precipitates obtained with 45 amines are tabulated.

L. A. O'NEILL

3017. **Rapid analysis of mixtures of methylamines and ammonia.** H. Guinot and —. Bellier (*Bull. Soc. Chim. France*, 1954, **21** [6], 727–732).—Rapid methods of analysing aq. solutions containing the three methylamines and NH_3 are investigated and a suitable procedure for determining all four components in a mixture is developed. (*i*) Addition of a 1 to 1½ mol. excess of formaldehyde to the soln. converts methylamine into 1:3:5-trimethyltrimethylenetriamine (**I**), b.p. 166° C, dimethylamine into bisdimethylaminomethane (**II**), b.p. 85° C, and NH_3 into hexamine, whilst trimethylamine is unchanged and can be removed completely by distillation of the mixture at 60° C and 25 cm pressure of water in a current of CO_2-free nitrogen (40 litres per hr.). After passing through two wash-bulbs containing formaldehyde, the trimethylamine is absorbed in water in an air-tight flask and titrated with H_2SO_4 to a methyl red end-point. (*ii*) Trimethylamine and **II** can be removed together (after addition of 20 g of KOH per 50 ml of soln.) by a similar distillation at atm. pressure and 70° to 80° C; the two bulbs should each contain *N* NaOH (5 ml) to retain any volatilised **I**. The dimethylamine content is calculated from the difference between the titrations in (*i*) and (*ii*). For max. accuracy the sample should contain $\lessapprox$ 5 to 10 per cent. of dimethylamine. (*iii*) Methylamine is determined by addition to the soln. (containing KOH) of a ten-fold excess of benzaldehyde (aq. emulsion) to ensure formation of benzylidenemethylamine (b.p. 180° C), followed by distillation, at $\simeq$ 75° C of di- and trimethylamines and NH_3, their absorption in H_2O and titration with H_2SO_4. The total alkalinity of the sample is also determined so as to obtain the methylamine content by difference. Results for various mixtures (0·1 to 0·6 mol. of each component per litre) are listed; the error is min. ($\pm$ 2·5 per cent.) for mono- and tri-methylamines, and except for very small amounts of dimethylamine is always less than 5 per cent. A complete analysis takes about 3 hr.

W. J. BAKER

3018. **Determination of acrylonitrile by means of the cyanoethylation reaction.** A. P. Terent'ev, P. F. Butskus and V. G. Yashunsky (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 162–165).—Acrylonitrile is determined by addition of a known amount of a solution of aminoacetic acid and evaluation of the excess by means of van Slyke's gasometric method. The method is suitable for pure acrylonitrile and for use with coloured solutions; water or ethylene cyanohydrin does not interfere. G. S. SMITH

3019. **Volumetric determination of mercury in phenylmercury bromide and in fungicidal preparations containing phenylmercury bromide.** K. Kámen (*Chem. Listy*, 1953, **47** [7], 1008–1013).—The method consists in the destructive oxidation of phenylmercury bromide (**I**) with $KMnO_4$ in H_2SO_4 and the titration of Hg^{++} as $HgSO_4$ with NH_4CNS. *Procedure*—Treat the sample of **I** (0·6 g approx.) with 75 per cent. H_2SO_4 (25 ml) and add gradually, in 8 portions (in approx. 8 min.), powdered $KMnO_4$ (2·5 g). After standing it at room-temp. for 15 min., heat the mixture on a steam-bath. Add $KMnO_4$ (3 g) in 6 portions during 2·5 hr., and continue heating for a further 30 min. By then the odour of bromine should have disappeared. Add conc. H_2SO_4 (5 ml), boil for 20 min., allow to cool, dil. to approx. 130 ml with H_2O and clarify with 6 per cent. H_2O_2 soln. Boil for 3 min. to destroy the excess of H_2O_2, dil. to 200 ml, cool to 12° C and titrate with 0·1 *N* NH_4CNS, using $NH_4Fe(SO_4)_2$ (2 ml of saturated soln.) as indicator. Phenylmercury bromide in fungicidal preparations is determined similarly. G. GLASER

3020. **Determination of diphenylmercury alone or in presence of phenylmercury compounds. Application to ethyl analogues.** V. L. Miller and D. Polley (*Anal. Chem.*, 1954, **26** [8], 1333–1335).—For the determination of diphenylmercury alone, it is extracted from a $CHCl_3$ soln. with 9 *N* HCl. Phenylmercury chloride is quant. formed and this is determined by reaction with dithizone and measurement of the colour produced at 620 mμ. Diethylmercury is determined in the same way except that 12 *N* HCl is used for the hydrolysis. For the determination of diphenyl- or diethylmercury in the presence of phenyl or ethylmercury compounds, the latter are removed by extraction into acidified $Na_2S_2O_3$ soln. and the diphenyl- or diethylmercury is determined as above. Beer's law is obeyed over the ranges 1 to 30 μg and 90 to 120 μg. The error is $<$ 5 per cent. G. P. COOK

3021. **Investigation of the Kokatnur - Jelling method for the determination of benzoyl peroxide.** A. J. Radford (*Analyst*, 1954, **79**, 501–504).—Owing to difficulties in obtaining reproducible results, the iodimetric method of Kokatnur *et al.* (*J. Amer. Chem. Soc.*, 1941, **63**, 1432) for determination of benzoyl peroxide was examined. The factors affecting the accuracy are detailed and a modified procedure is recommended in which $\simeq$ 0·5 g of benzoyl peroxide is added to 50 ml of *iso*propanol in a tall-form beaker and treated with 5 ml of a saturated aq. soln. of KI and 5 ml of glacial acetic acid. The covered beaker is heated for 2 to 3 min. at "incipient" boiling. Without cooling, the liberated I is titrated with 0·1 *N* $Na_2S_2O_3$. The heating procedure is the most important factor and must be regulated so that uncondensed vapour does not reach more than half-way up the sides of the beaker. A. O. JONES

3022. **The identification of the nitrophenols as their aryloxyacetic acids.** J. Grundy (*Analyst*, 1954, **79**, 523–524).—Conversion of compounds containing the phenolic group into the corresponding aryloxyacetic acids is an established means for their identification. A number of workers have failed to condense the 2- and 4-nitrophenols with ethyl chloroacetate by suitable methods. It has been confirmed that failure was due to temp. conditions. A method has been evolved for rapid preparation of the nitrophenoxyacetic acids in effectively quant. yields. Sodium (0·005 g-atom) is dissolved in 2 ml of anhydrous methanol by heating it in a 5-ml flask with a reflux condenser in an oil-bath, the nitrophenol (0·005 mole) in 1 ml of methanol is added and the apparatus is set for distillation. The temp. of the oil-bath is raised to and maintained at 100° to 140° C until all the methanol is removed, triethylene glycol (3 ml) is added and heating is continued until all solid sodium has dissolved. A slight excess of ethyl chloroacetate or a higher ester is added and the mixture is heated at 180° to 200° C for 30 min. Finally the hot mixture is poured into 20 ml of 10 per cent. w/w H_2SO_4 and hydrolysed under reflux. The clear soln. is cooled and the crystalline acid is collected, washed with cold water and dried. A. O. JONES

3023. **The quantitative chromatographic separation of ortho- and para-nitroanilines and their monosubstituted derivatives.** J. E. Larson and S. H. Harvey (*Chem. & Ind.*, 1954, [2], 45–46).—A solution of isomeric nitroanilines (1 g) in reagent-grade benzene (75 ml) was applied to a column of activated alumina and the chromatogram was developed with 20 per cent. ethyl acetate in benzene. Results were best with columns prepared by sedimentation. The ortho isomer formed an orange band at the bottom of the column and was eluted with 20 per cent. ethyl acetate in benzene, whilst the para isomer formed a yellow band at the top of the column and was eluted with 40 per cent. ethyl acetate in benzene; the meta isomer when present formed a pale yellow band between the two. The isomers were estimated gravimetrically after removal of the solvent by distillation and drying to constant weight *in vacuo*. A number of substituted nitroanilines can be resolved in this way. D. LIFF

3024. **General method for the determination of diazo compounds and in particular "fast salts" (stabilised diazo compounds of aromatic amines without solubilising groups).** L. Nicolas, P. Lampel and J. Planté (*Chim. Anal.*, 1954, **36** [9], 238–241).—In a flask with a central neck and 3 tubulures, 150 ml of 30 per cent. H_2SO_4 and 0·5 g of Cu_2Cl_2 is boiled to remove air and cooled. The central neck is provided with a reflux condenser connected to a nitrometer filled with 40 per cent. KOH soln., and the tubulures are used for a supply of CO_2 (from heated $NaHCO_3$), a thermometer, and for a glycerined glass rod from which is suspended sufficient of a weighed sample of the diazo-compound to give 20 ml of N. Air is driven from the apparatus with CO_2, and, after connection with the nitrometer, the sample is immersed in the H_2SO_4, which is then boiled until liberation of N is complete. The N is withdrawn from the nitrometer into a graduated tube by means of a cup fitted at its top, and its vol. is measured over water. Examples are given of the results of the method in comparison with other methods of analysis. E. J. H. BIRCH

3025. **Displacement analysis of terpene hydrocarbons. I. Preliminary studies.** A. C. Glasser, L. E. Harris, B. V. Christensen and F. W. Bope (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [5], 294–296).—From experiments on α-pinene, limonene, toluene and ethyl acetate, it is shown that in displacement analysis on silica gel adsorption isotherms may be used to predict the nature of the displacement diagrams. When 25 per cent. toluene in *n*-hexane is used as a displacer, limonene appears as a zone in the analysis of a simple mixture of limonene and α-pinene, but with more complex mixtures of natural terpenes separation is not effected. E. HAYES

3026. **Determination of pyridine bases in the surface of aluminosilicate catalysts.** K. G. Miesserov (*J. Appl. Chem., U.S.S.R.*, 1954, **27** [5], 566–567).—The aluminosilicate is mixed with 15 per cent. aq. alkali, and the pyridine bases are distilled off, trapped in water and titrated directly with 0·029 *N* HCl with bromophenol blue as indicator. The max. error does not exceed 1 per cent. R. C. MURRAY

3027. **Analysis of silico-organic compounds. Qualitative analysis of mixtures of alkoxysilanes with alcohols and silicon tetrachloride.** A. P. Kreshkov, V. A. Bork and V. T. Shemyatenkova (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [3], 166–169).—The presence of silicon tetrachloride in tetramethoxy- and tetraethoxy-silanes can be detected by (*a*) reaction with HI to give I, (*b*) reaction with aniline or with an aniline solution of tetramethyldiaminobenzophenone to give a white or yellow ppt. sol. in excess of reagent, (*c*) reaction with water to give HCl, (*d*) reaction with $K_2Cr_2O_7$ and nitric acid in parallel with a blank containing the esters only, whereby the slower formation of a blue-violet colour indicates the presence of $SiCl_4$, or (*e*) reaction with ferric ferrihexathiocyanate, $Fe[Fe(SCN)_6]$, to give decolorisation. The presence of methanol in tetramethoxy-silane and ethanol in tetraethoxy-silane can be detected by (*a*) reaction with Na to give H, (*b*) reaction with $Fe[Fe(SCN)_6]$, whereby a red solution is obtained in presence of an alcohol, or (*c*) reaction with $SiCl_4$ to give HCl. In presence of $SiCl_4$ only, the esters can be detected by their reaction with borax and HCl or H_2SO_4, followed by a flame test. In presence of the alcohols only the esters can be detected by the formation of SiO_2 on being heated with HCl. G. S. SMITH

3028. **Detection of periodate-oxidisable compounds on paper chromatograms.** R. L. Metzenberg and H. K. Mitchell (*J. Amer. Chem. Soc.*, 1954, **76** [16], 4187).—The chromatogram prepared on Whatman 1 filter-paper (preferably washed initially with the solvent used) is dried thoroughly to remove residual acid or base. It is then sprayed very lightly and evenly with aq. 0·01 *M* KIO_4, dried at $\simeq$ 18° C for 10 min. and then sprayed with a soln. of 35 per cent. $Na_2B_4O_7$ containing 0·8 per cent. KI, 0·9 per cent. boric acid, and 3 per cent. sol. starch. Over areas where periodate has been reduced to iodate (*e.g.*, by glycols or nucleosides) white spots are seen on the blue background of non-reduced areas. Optimum contrast is obtained after $\simeq$ 10 min., and the non-permanent spots can be photographed. Phenolic or buffered solvents interfere. The degree of specificity of 70 compounds is reported; the method is sensitive to 0·25 μg of inositol or its equiv. in periodate reducing power. Usefulness is limited by the reaction of periodate with the paper. W. J. BAKER

3029. **The determination of elementary sulphur in petrol by Sommer's test.** A. Albert (*Erdöl u. Kohle*, 1954, **7** [5], 289–291).—Results obtained with variations of Sommer's test—detection of elementary S by colour formation on adding alkali to S dissolved in pyridine—are described. When the test is carried out according to Mapstone's method (*Brit. Abstr. C*, 1946, 238 and 246), the results are negative if the petrol has been purified with reducing agents such as sulphide or bisulphite. The presence of formaldehyde, formic acid and olefins in the petrol also causes negative results. Although Mapstone's test does not fail with petrol purified by conc. H_2SO_4, it fails with samples purified by plumbite or hypochlorite. To ascertain whether the petrol has been treated with plumbite or hypochlorite, a sample is shaken with $NaHSO_3$ and then with NaOH. If pyridine is then added and the aq. alkaline phase turns red-violet, hypochlorite or plumbite has been used. The author gives a procedure for the detection of amounts of S smaller than those detectable by Mapstone's test. With the author's procedure, $FeSO_4$ and Na formate prevent the colour formation, whereas Na_2S in alkali both stabilises and intensifies the colour. In presence of $Fe^{...}$, the colour is at first prevented from being formed, but it appears on long standing and is then stable for more than 48 hr.

C. J. KEATTCH

3030. **A photometric method for the estimation of the oil yield of oil shale.** F. Cuttitta (*U.S. Geol. Survey Bull.*, 1953, No. 992, 15–31).—Shale (0·5 g) crushed to — 80 mesh is weighed into a 25-ml Pyrex glass-stoppered test tube. The test tube is heated in a horizontal position in a special tubular furnace at 480° to 500° C for 11 to 14 min. At the completion of the distillation, the test tube is removed and cooled in air to room temp., 10 ml of toluene are added and the tube is shaken. The liquid is filtered and the filter-paper and residue are washed twice with 5-ml portions of toluene. The combined extract and washings are diluted to 25 ml with toluene. The optical density (× 10) of the soln. is measured at 420 mμ (blue filter) on a filter photometer with pure toluene set at 100 per cent. transmission. Oil yield is determined from a standard curve.

CHEM. ABSTR.

3031. **A volumetric method for the estimation of the oil yield of oil shale.** F. Cuttitta (*U.S. Geol. Survey Bull.*, 1953, No. 992, 33–37).—A 1-g sample of — 80 mesh shale is weighed into a test tube. The lower third of the glass-stoppered test tube is heated in a horizontal position in a special tubular furnace at 480° to 500° C for 11 to 14 min. After cooling, ethyl acetate (10 ml) is added and the tube is well shaken. The soln. is filtered and washed twice with 3-ml portions of ethyl acetate. The extract and washings are combined in a centrifuge tube and heated in a steam bath to volatilise the ethyl acetate. The residue is centrifuged while still hot and the vol. of oil is read. On assuming an average sp. gr. of 0·903, gallons of oil per ton of shale = 239·3 × (ml of oil).

CHEM. ABSTR.

3032. **The testing of lubricants.** A. v. Hoyningen-Huene (*Erdöl u. Kohle*, 1954, **7** [6], 393–397).—Standard tests are reviewed. Colour, density, viscosity, viscosity-index, carbonisation, flash-point, solidification-point, ash content, water content, tendency to emulsify, neutralisation number, saponification number, foam-test, volatility and ageing are discussed.

E. HAYES

3033. **Lüers method of cyclic extraction in the analysis of mineral oils.** E. H. Kadmer (*Brennstoff-Chem.*, 1954, **35** [11–12], 170–175).—After oils have been fractionally extracted by continuous percolation with methanol, the extract being withdrawn in successive cuts, the first cut contains mainly aromatics, with some lower naphthenes and paraffins, and the O and S compounds. The selectivity of the methanol for aromatics is enhanced by the presence of $\simeq$ 6 per cent. of water. The aromatic content falls in succeeding cuts and naphthenes are preferentially extracted to leave finally a paraffinic residue. Characteristics of the fractions so obtained from various petroleum and raffinates and their location in the aromatic-naphthene-paraffin triangle, are recorded. The higher alcohols are progressively less selective than methanol, but are useful for cylinder oils only slightly soluble in methanol.

A. R. PEARSON

3034. **Evaluation of chemical de-icing solutions.** C. Berger (*Bull. A.S.T.M.*, 1954, No. 197, 67–68).—A formula expressing the effectiveness of a de-icing spray is given, and a method for determining the only unknown variable, C, is described. The procedure involves freezing 10 ml of H_2O at 0° C in a Petri dish, covering the ice surface evenly with 5 ml of solvent at 0° C, keeping the dish and contents at 0° C for $\simeq$ 3 min., and determining the increased vol. of liquid subsequently decanted and measured. Values of C (wetting and ice-dissolving power of compound) are expressed for 100 ml of H_2O per sq. cm per min., and are highest for 2-amino-2-methylpropan-1-ol (8·3), 2-methoxyethanol (7·6), acetone (5·9), glycerol and *iso*propanol (4·1). Reproducibility for tests on any one solvent is ± 0·15 ml.

W. J. BAKER

3035. **Quantitative field tests for quaternary ammonium compounds.** C. C. T. Chinnick and P. A. Lincoln (*Lab. Practice*, 1954, **3** [9], 364–366).—Two titrimetric methods for determining quaternary ammonium salts were investigated and one was adapted for field use. Methylene blue is displaced from its $CHCl_3$-soluble salt of an alkyl sulphate by the quaternary compound. Twenty ml of a standard solution of sodium dodecyl sulphate, tetradecane-2-sulphate or octyl sulphosuccinate are transferred to a measuring cylinder and 25 ml of aq. indicator (0·03 g of methylene blue, 12 g of H_2SO_4 and 50 g of Na_2SO_4 in 1 litre) and 15 ml of $CHCl_3$ are added. Quaternary salt (0·005 N) is added until the $CHCl_3$ and aq. phase have the same colour by reflected light. The method is neither stoicheiometric nor generally applicable and must be standardised for each compound. The quaternary ammonium salts of bromophenol blue are soluble in ethylene dichloride, but the Na salt is not. Twenty ml of one of the above alkyl anionic solutions (0·005 N) are placed in a measuring cylinder and 25 ml of aq. indicator (0·04 g of bromophenol blue, 100 g of NaCl and 10 g of Na_2CO_3 in 1 litre), 25 ml of water and 20 ml of ethylene dichloride are added. The mixture is titrated with 0·005 N quaternary ammonium salt until the previously colourless organic layer is deep blue. Results by this method are nearly stoicheiometric, and it is adapted for the field test in which in a 25-ml cylinder are placed 3 ml of ethylene dichloride, 5 ml of indicator solution and 2 ml (from a calibrated dropper) of 0·001 N sodium octyl sulphosuccinate. The vol. of quaternary ammonium salt solution required to turn the organic layer blue (total cylinder contents less 10 ml) is a measure of its concn. Results for

cetyltrimethyl- and didecyldimethyl-ammonium bromides and cetyl- and lauryl-pyridinium chlorides are given for the field test. E. J. H. BIRCH

3036. **Detection and estimation of melamine in wet-strength paper by ultra-violet spectrophotometry.** R. C. Hirt, F. T. King and R. G. Schmitt (*Anal. Chem.*, 1954, **26** [8], 1273–1275).—The sample of paper is refluxed with 0·1 *N* HCl to remove the melamine resins and to hydrolyse them to melamine (2 : 4 : 6-triamino-*s*-triazine). The absorption of the extract is measured at 235 mμ and corrected for background by subtracting the absorption at 260 mμ. The method gives results in good agreement with the Kjeldahl - nitrogen procedure, average differences being 5·5 per cent. relative. The sensitivity is 4 μg. G. P. COOK

3037. **Determination of menthone, menthol and menthofuran in peppermint oil. Localisation of menthofuran in the plant.** J. A. J. M. Lemli (*Ann. Pharm. Franç.*, 1954, **12** [4], 275–281).—For menthone, 25 mg of the essential oil are dissolved in 10 ml of $CHCl_3$ and 1 ml of the solution is diluted (10 + 1) with methanol. One ml of dinitrophenylhydrazine reagent (0·1 per cent. w/v) and 1 ml of aq. HCl - methanol (1 + 100 by vol.) are added and the mixture is heated for 75 min. at 55° C. After cooling, 5 ml of a mixture of aq. KOH (10 ml), H_2O (10 ml) and methanol (up to 100 ml) are added and the absorption is measured, after 8 min, at 535 mμ. Free menthol is determined by oxidising 25 to 30 mg of oil with 0·5 ml of a solution of $K_2Cr_2O_7$ (13 g), conc. H_2SO_4 (16 g) and H_2O (60 g) for 2 hr. The excess of CrO_3 is decomposed with methanol in 15 min. The menthone formed is extracted with $CHCl_3$ and determined as before. Total menthol is similarly determined after first saponifying 25 mg of oil with 0·5 *N* KOH - methanol at 65° C for 2 hr., and cooling before the oxidation. For menthofuran the method of Krupski and Fischer (*Brit. Abstr. C*, 1951, 12) is modified by treating the oil in $CHCl_3$ with trichloroacetic acid - $CHCl_3$ and measuring the absorption at 500 mμ after 1 hr. The percentage of menthofuran in the oil obtained from different parts of the plant, and from whole plants at different stages of development is stated. E. J. H BIRCH

3038. **The absorptiometric determination of a non-ionic detergent.** D. G. Stevenson (*Analyst*, 1954, **79**, 504–507).—Two procedures for the determination of a non-ionic detergent are described, both involving pptn. by molybdophosphoric acid and subsequent absorptiometric determination of a soln. of the ppt. In one, the ppt. is dissolved in conc. H_2SO_4 and the absorption of the violet to rose-pink coloured soln. is determined at 520 mμ. The concn. of the detergent is then calculated by reference to a previously constructed calibration graph. In the other method, which is suitable for higher concn. and makes unnecessary the use of conc. H_2SO_4 in the absorptiometer cell, the ppt. is dissolved in conc. H_2SO_4, the soln. is diluted, treated with NH_4CNS and $SnCl_2$ and the absorption of the amber soln. is determined at 470 mμ. The second method is the more sensitive. Interference by alkylarylsulphonates is negligible. Alkyl sulphate and carboxymethylcellulose interfere, apparently by acting protectively on the ppt. A. O. JONES

3039. **Tentative methods of test for wax polishes.** A.S.T.M. Committee D-21 (*Bull. A.S.T.M.*, 1954, No. 197, 38–40).—The proposed apparatus and procedures for (*i*) concentrating the extenders, and (*ii*) determining the refractive index of carnauba wax or other high m.p. natural and synthetic waxes are specified. The $CHCl_3$ extract of the additives contains certain components in greater amounts than existed in the original wax, so that the index properties of this extract (sap. val., acid val., n_D^{100}) greatly facilitate the detection of extenders and contaminants in floor polishes. Refractive indices of carnauba wax extenders are listed. W. J. BAKER

3040. **Turbidimetric titration of polyvinyl chloride.** A. Oth and V. Desreux (*Bull. Soc. Chim. Belg.*, 1954, **63** [5–7], 261–284).—Turbidimetric titration of polyvinyl chloride has been carried out with *cyclo*hexanone as solvent, and heptane - CCl_4 (9 + 1) as precipitant. To obtain a measurement proportional to the concn. of pptd. polymer, it is necessary to record the scattered light at 3 angles (45°, 90° and 135°) and to correct for changes of particle dimensions during titration. Solubility curves have been obtained for several polymers and fractions therefrom. Relations between the vol. fraction of precipitant (volume of precipitant divided by the sum of volumes of solvent and precipitant), polymer concn. and mol. wt. have been studied. It is possible to derive approx. mol. wt. distribution curves. L. A. O'NEILL

3041. **Determination of hexamethylenetetramine [hexamine] in industrial phenol - formaldehyde resins.** P. Hamard and L. Jacqué (*Chim. et Ind.*, 1954, **71** [5], 915–918).—The usual method of determining hexamine (**I**) in moulding powders by titration of aq. extracts of the powder with 0·1 *N* H_2SO_4 (*i*) with phenolphthalein (to give the NH_3 content) and (*ii*) with methyl orange (to give NH_3 plus **I**) is liable to error, owing to loss of NH_3 during grinding, the doubtful end-point with methyl orange and the instability and complexity of the aq. extracts. The N in phenol - formaldehyde resin products is present as NH_3, as organic bases soluble in water and chemically bound in mol. macromolecules; it is proposed that these three fractions should be analysed separately by determining (*i*) total N by a Kjeldahl semi-micro determination on a 100 to 200-mg sample, (*ii*) soluble N by Kjeldahl semi-micro determination on the aq. extract from a second sample obtained by agitation with water for 3 hr. at room temp., and (*iii*) chemically-bound N by Kjeldahl on the residue from the aq. extraction. Results obtained by this fractional analysis are in reasonably good agreement with the total N content obtained by direct analysis. J. M. JACOBS

3042. **Estimation of rubber in asphalt.** G. Salomon, E. Pezarro - van Brussel and A. C. van den Schee (*Anal. Chem.*, 1954, **26** [8], 1325–1328).—Raw or vulcanised, natural or synthetic rubber is separated from the inorganic matter of the asphalt by prolonged extraction (48 hr.) with xylene or solvents having higher boiling points. The solvent is removed by evaporation and the rubber is treated with twice its wt. of sulphur at 155° C for 4½ hr. The ebonite fraction is extracted and purified, and the rubber content is calculated from a sulphur determination on the purified ebonite. Influence on the reaction of time, temp., the type of rubber and bitumen and the viscosity of the mixture has been studied. Results were from 10 to 20 per cent. high on bitumen - rubber mixtures containing from 2 to 10 per cent. of rubber. If the asphalt has been heated above 265° C before analysis, most of the rubber is lost. G. P. COOK

3043. **Hardness testing of elastomers at low temperatures.** B. G. Labbe (*Bull. A.S.T.M.*, 1954, No. 199, 73–79).—The performance of seven different hardness testers (Durometer, Indentometer and Plastometer types) was compared with respect to efficiency and reproducibility at low temp. (+ 60° to − 80° F) for several rubber-like materials. The spring-actuated instruments giving instantaneous readings are generally preferable to dead-weight loading devices, some of which are often difficult to operate at − 60° to −80° F owing to the manner of loading and to friction (often caused by frosting) between the moving parts. Use of a vibrator reduces friction and ensures more reproducible results. Some precautions for low-temp. operation are given. W. J. Baker

3044. **Stiffness testing of elastomers at low temperatures.** F. S. Conant (*Bull. A.S.T.M.*, 1954, No. 199, 67–73).—The variations in Young's modulus at low temp. of rubber-like compounds (Hevea gum, GRS tread-type, and polyethylene) as determined in 37 different laboratories by different test procedures such as the tension-modulus, cantilever-beam, simple-beam, and torsion methods are reported. Correlation between values obtained by the simple-beam method was closest, although these values are not necessarily the truest. Over the critical range of temp. influencing technical practice (*viz.*, when the rubber is changing from the flexible to the hard and stiff state), the different tests show good agreement. In reporting the modulus of elastomers and plastics, the method of obtaining that value should also be given.
W. J. Baker

3045. **Quantitative analysis of polyisoprene by means of infra-red spectra.** E. I. Pokrovskii and M. V. Vol'kenshtein (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1954, **95**, 301–303).—An i.r. analytical procedure is outlined which permits quant. determination of relative amounts of polyisoprene, fractions containing the C:C bond in the *cis*-1:4- and *trans*-1:4-, 1:2- and 3:4- positions in synthetic polyisoprenes. Samples of polyisoprene are analysed (their optical density being determined at 815, 839, 853, 888 and 909-cm^{-1} bands) with natural rubber, guttapercha, hept-1-ene and 2:3:3-trimethylbut-1-ene as model compounds for the *cis*-1:4-, and *trans*-1:4-, 1:2- and 3:4-fractions (in the given order). More accurate results are claimed than those obtained by the recent method of Richardson and Sacher (*J. Polymer Sci.*, 1953, **10**, 353).
S. K. Lachowicz

3046. **Effect of sample preparation on precision in the hide substance determination.** S. Dahl (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 515–518).—The American Leather Chemists' Association method of sample preparation for hide substance determination is inadequate for chrome-tanned highly-greased upper leather. Precision is improved by a second grinding, but not by increasing specimen size from 0·5 g to 1·5 g. The reason for this is discussed. It is recommended that the A.L.C.A. method be revised so that the sample is passed several times through a Wiley mill, preferably with a 2-mm sieve. B. R. Hazel

3047. **Sampling of vegetable-tanned leathers. American Leather Chemists' Association, Provisional Method, May, 1954.** (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 466–468). The method for sampling all types of vegetable-tanned leather and products made from it as a preliminary to analysis is stated.
B. R. Hazel

3048. **Methods for the analysis of vegetable-tanned leathers. American Leather Chemists' Association Provisional Methods, May, 1954.** (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 468–490).—Methods are given for moisture analysis, determination of material extractable in ether and chloroform, and determination of total N and hide substance, water-soluble material, soluble non-tannin and uncombined tannin, glucose, insoluble ash, combined tannin and degree of tannage, total ash, Mg as Epsom salt, pH, and Procter and Searle number (for H_2SO_4). B. R. Hazel

3049. **Method for the analysis of mineral tanning materials. American Leather Chemists' Association Provisional Methods, May, 1954.** (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 490–493).—The analytical method is given in detail for determining Cr_2O_3, acid and basicity in one-bath chrome liquors.
B. R. Hazel

3050. **Sampling of mineral-tanned leathers for chemical tests. American Leather Chemists' Association Provisional Methods, May, 1954.** (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 493–495).—Directions are given for sampling all types of mineral-tanned leather and for preparation of composite samples for each test. B. R. Hazel

3051. **Methods for the analysis of mineral-tanned leather. American Leather Chemists' Association Provisional Methods, May, 1954.** (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [7], 495–505).—Methods are given in detail for analysis of mineral-tanned leather for Cr_2O_3, total, neutral and combined acid SO_4'', total Cl', sulphate, basicity and pH.
B. R. Hazel

See also Abstract 2912.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

3052. **Identification of organic acids in urine by paper chromatography.** R. Nordmann, O. Gauchery, J.-P. du Ruisseau, Y. Thomas and J. Nordmann (*Compt. Rend.*, 1954, **238** [25], 2459–2461).—Non-ionised compounds, ampholytes, cations and chlorides are first removed by filtering the urine sample (5 ml) plus water (50 ml) through a 10-cm column of Dowex-2 resin (converted to formate) with 6 *N* formic acid (50 ml) as eluting solution for the organic acids. Eluate is evaporated to dryness at 100° C, the residue is dissolved in water (100 ml), and the solution is chromatographed (two-dimensional ascending procedure) from one corner of a Whatman No. 1 filter-paper (40 cm square), by the successive use of ethanol - aq. NH_3 - water (16:1:3 by vol.) and propanol - cineole - formic acid (5:5:2 by vol.) saturated with H_2O. The spots are revealed with a soln. of bromocresol green in ethanol, and are identified by reference partly to standard spots and partly to differential R_F values (*Bull. Soc. Chim. Biol.*, 1952, **34**, 380). In addition to acetic acid (volatised during the initial evaporation), the acids identified as above in normal urine are: aconitic, citric, glutaric, α-oxoglutaric, glycollic, hippuric, lactic and β-hydroxybutyric (incompletely separated), malic, succinic, tartaric, and an unknown phenolic acid. Three spots are as yet unidentified.
W. J. Baker

3053. **On the presence of taurocyamine (guanidotaurine) in the urine of the rat and its biochemical significance in the excretion of nitrogen.** Nguyen-van Thoai, J. Roche and A. Olumucki (*Biochim. Biophys. Acta*, 1954, **14** [3], 448).—The isolation and identification of taurocyamine has been carried out by the following method. Urine is passed through a 28 × 400-mm column of Permutit C50, which fixes cations and arginine. The eluate, concentrated under vacuum, is filtered through Permutit A300. The effluent liquid is subjected to ascending paper chromatography with the solvents; pyridine - *iso*pentanol - acetic acid - water (80 : 40 : 10 : 40), butanol - acetic acid - water (73 : 10 : 17), pyridine - *iso*pentanol - aq. NH_3 (20 per cent.) - water (80 : 40 : 10 : 40), and propanol - aq. NH_3 (20 per cent.) - water (73 : 20 : 7). Synthetic taurocyamine was run as control and the spots were developed with sodium-1-naphthol hypobromite.

G. W. CAMBRIDGE

3054. **Bedside determination of total base in serum.** B. H. Scribner and H. T. Wiegert (*J. Amer. Med. Ass.*, 1954, **155** [7], 639–644).—A rapid simple method requiring 0·25 to 0·5 ml of serum is described. Results are calculated from the increase in titratable acidity obtained on passing the sample through a cation-exchange resin after correction for bicarbonate content, which is determined separately (*Anal. Abstr.*, 1954, **1**, 2477). The precision is ± 0·5 milli-equivalents per litre and the standard deviation of the mean is 1·3 milli-equivalents per litre. *Reagents*—reagents (*i*) and (*iii*) are as described previously (*loc. cit.*). Standardise (*i*) against 0·1 *N* acid. (*iv*) Sieve 20 to 50-mesh (Tyler) Dowex 50 or Amberlite 120 resin cross linked with 8 or 9 per cent. divinylbenzene on a 35-mesh (Tyler) sieve. Treat 1 vol. of the material passing the sieve with ≮ 20 vol. of 4 per cent. H_2SO_4 passed slowly through during 24 hr. Wash the prepared resin free from acid and store under water. *Procedure*—Place a small disc of 70 to 80-mesh wire screen inside the barrel of a 1-ml tuberculin syringe and fill the barrel (containing a little water) with wet resin to between the 0·5 and 0·6-ml marks. Allow to drain. Discharge the standard vol. (0·5 ml—see below) of sample from a 1-ml tuberculin syringe on to the resin column and wash through with two 0·75-ml amounts of distilled water, collecting the eluate in a 1-in. × 4-in. Pyrex-glass test tube. Add six drops of 0·04 per cent. aq. phenol red, and swirl for 2 min. to remove CO_2. Titrate slowly, with the needle-tip below the surface, from a syringe charged with 1 ml of the standard NaOH reagent (*iii*) until the colour matches that of 2·5 ml of buffer (pH 7·4) containing 0·25 ml of indicator. The vol. of NaOH in ml used multiplied by 200 plus the serum bicarbonate content in milli-equivalents per litre gives the total base in milli-equivalents per litre. Standardise the procedure with the standard vol. (approx. 0·5 ml) of (*i*) and adjust this vol. if necessary so that 0·50 ml of (*iii*) is required in the titration. The method is subject to errors similar to those of the bicarbonate determination (*q.v.*).

W. H. C. SHAW

3055. **A rapid method for filter-paper electrophoresis.** S. D. Vesselinovitch and H. S. Funnell (*Canad. J. Biochem. Physiol.*, 1954, **32** [5], 567–570).—A plastic container (30 × 20 × 4 cm) divided lengthwise into four equal compartments that contain the buffer is placed in a plastic covered dish (23 × 23 × 10 cm). The two inner compartments of the container carry platinum foil electrodes and are connected to the outer compartments by bridges of cellulose tissue. The paper strips are supported on nylon bristles cemented to a plastic plate (27 × 15 cm) held in position above the buffer vessel by two parallel rods fixed across the main chamber. Strips of Whatman No. 1 filter-paper (28 × 4 cm) are saturated with barbiturate buffer pH 1·6, ionic strength 0·05, laid upon a paper towel to remove any excess of buffer and hung across the nylon bristle support, which is then placed in the chamber so that the ends of the strips dip a few mm into the buffer in the outer compartments. Serum (0·005 to 0·01 ml) is applied evenly along a line 1·5 cm from the centre of the strip toward the negative pole, the chamber is closed and the current switched on for 2 hr, a potential gradient of 12 to 14 V per cm being used. The strips are then removed, dried for 10 min. at 120° C, stained for 5 min. with 1 per cent. bromophenol blue in ethanol saturated with $HgCl_2$, washed with 0·2 per cent. acetic acid until no further yellow colour is removed and finally immersed in absolute ethanol for 2 min. The rapidity and distinctness of separation of the protein fractions depends chiefly upon the choice of potential gradient, ionic strength of buffer, and small amount of serum used. H. F. W. KIRKPATRICK

3056. **Examination of the method of paper electrophoretic serum analysis.** W. Grassmann and K. Hannig (*Klin. Wschr.*, 1954, **32** [35–36], 838–846).—The factors influencing the detection, separation and estimation of serum proteins by paper electrophoresis are critically analysed. G. W. CAMBRIDGE

3057. **Studies on paper electrophoresis.** H. K. Oosterhuis (*J. Lab. Clin. Med.*, 1954, **44** [2], 280–291).—Paper electrophoresis of serum proteins as an aid to clinical diagnosis is preferred to the chemical method of Gleiss and Hinsberg (*Brit. Abstr. C*, 1951, 298), with which comparisons are made. The construction of the electrophoresis apparatus and of a scanning device for use with the stained paper strips are described in detail. Electrophoresis patterns obtained with normal and with a number of abnormal sera are given, and the correlation of the patterns with various pathological conditions is discussed. W. H. C. SHAW

3058. **Improvement in the paper electrophoresis of serum proteins: decolorisation of strips by Kumagawa extraction.** B. Drevon (*Bull. Soc. Chim. Biol.*, 1954, **36** [6–7], 921–923).—Paper strips after electrophoresis and dyeing of proteins with Amido black 10 B are wound on a nylon apron as used for developing photographic film, and placed in the syphon chamber of a Kumagawa extraction apparatus. Complete decolorisation of the background is attained in 30 to 45 min. with 3 to 4 changes of solvent [a (9 + 1) mixture of methanol and acetic acid] with considerable saving of time and solvent and without loss of protein. C. E. SEARLE

3059. **The use of Amido black B for the detection of protein fractions obtained by paper electrophoresis.** R. Wolff and P. Magnin (*Bull. Soc. Chim. Biol.*, 1954, **36** [6–7], 925–926).—When the methanol-acetic acid mixture used for removing excess of Amido black B (Amidoschwarz B) after paper electrophoresis and staining of proteins is recovered by treatment with charcoal, the accumulation of barbitone buffer in the mixture results in some decolorisation of the stained proteins. After electrophoresis, therefore, the proteins are fixed by $HgCl_2$ and the buffer is removed by a stream of

water, the proteins then being stained in the usual way. The accumulation of buffer in the staining solution is not harmful. C. E. SEARLE

3060. **The determination of serum acid and alkaline phosphatase activity with 4-aminoantipyrin.** M. E. A. Powell and M. J. H. Smith (*J. Clin. Pathol.*, 1954, **7** [3], 245–248).—*Alkaline phosphatase*—To 2·0 ml of Na_2CO_3 - $NaHCO_3$ buffer and 2·0 ml of diphenyl phosphate soln. at 37° C, add 0·2 ml of serum. Incubate at 37° C for 15 min., and then add 2·0 ml of 0·1 *M* Na_2CO_3, 1·0 ml of 1·5 per cent. w/v 4-aminoantipyrin [4-aminophenazone] hydrochloride (**I**) and 1·0 ml of 4 per cent. w/v $K_3Fe(CN)_6$. Set aside at room temp. for 5 min. and read the colour on a Spekker absorptiometer with No. 604 green filters against a control in which serum is added after incubation. *Acid phosphatase.*—To 2·0 ml of citrate buffer and 2·0 ml of diphenyl phosphate soln. at 37° C, add 0·2 ml of serum. Incubate at 37° C for 60 min., then add 2·0 ml of a soln. containing 4 per cent. w/v of Na_2CO_3 and 4 per cent. w/v of $NaHCO_3$, 1·0 ml of 1·5 per cent. w/v **I**, and 1·0 ml of 4 per cent. w/v $K_3Fe(CN)_6$. Set aside at room temp. for 15 min. and measure the colour as for alkaline phosphatase. Calibration curves are constructed by substituting 2 ml of soln. having various concn. of phenol (5 to 100 μg per ml) for the substrate, 1 μg per ml representing one King - Armstrong unit under the test conditions. Each soln. used for calibration had 0·2 ml of serum added, as serum depresses the optical density by 5 to 10 per cent. The Na_2CO_3 - $NaHCO_3$ buffer, citrate buffer and diphenyl phosphate soln. are those recommended by King ("Micro-Analysis in Medical Biochemistry," Churchill, London, Second Edition, 1951). The results showed good agreement with those given by King's method and the suggested method has the advantage of eliminating the necessity for pptn. of protein. H. F. W. KIRKPATRICK

3061. **Analysis of residual nitrogen compounds with the aid of the high-tension pherogram (a new analytical method). I.** L. Heilmeyer, R. Clotten, I. Sano, A. Sturm, jun., and A. Lipp (*Klin. Wochschr.*, 1954, **32** [35–36], 831–837).—A modification of Michl's method (*Mh. Chem.*, 1951, **82**, 489; 1952, **83**, 737) for the high-tension paper electrophoretic separation of amino-acids is described for the analysis of the non-protein nitrogenous fractions of serum, extra-cellular fluids and urine. Paper is suspended in hexane, instead of toluene, and the ends dip into electrode vessels containing a mixture of pyridine, acetic acid and water (1 : 10 : 90) at pH 3·6. Platinum electrodes are used and 1500 to 6000 volts are applied for 30 to 90 min. The proteins are removed from serum, exudates, etc., by treatment with methanol - acetone (3 + 1) and centrifuging, after setting the mixture aside for 30 min. at 0° C. Urine is freed from protein if necessary and de-salted by ion-exchange. The test solutions are concentrated by evaporation at atmospheric pressure and the residue is taken up in aq. methanol. Amounts equivalent to 1 ml of serum or 5 ml of urine are used for analysis. Spots are developed with ninhydrin and other specific agents such as *p*-dimethylaminobenzaldehyde for urea, $AgNO_3$ for uric acid and Na picrate for creatine and creatinine. G. W. CAMBRIDGE

3062. **Determination of deoxycholic and cholic acids in bile.** E. H. Mosbach, H. J. Kalinsky, E. Halpern and F. E. Kendall (*Arch. Biochem. Biophys*, 1954, **51** [2], 402–410).—The u.v. absorption method of Kier (*J. Lab. Clin. Med.*, 1952, **40**, 755) is modified in that, after hydrolysis with NaOH, extracts of the samples are heated in 65 per cent. H_2SO_4 to 60° C for 15 min. and cooled before the extinction values are measured. Under these conditions, cholic acid has max. absorption at 320 mμ and deoxycholic acid at 385 mμ. At these two wavelengths extinction varies as concentration for both compounds and the amount of each is calculated by a differential method. The procedure gives satisfactory recovery of both compounds but does not differentiate between deoxycholic and chenodeoxycholic acids. Comparable absorption curves are also given for sodium taurocholate, lithocholic acid and α-hyodeoxycholic acid. W. H. C. SHAW

3063. **Some reactions for the detection and determination of dehydrocholic acid.** C. Bergamini and W. Versorese (*Sperimentale*, 1954, **4** [5–6], 79–84).—The absorption at 530 mμ of the red solution produced by the action of *m*-dinitrobenzene on dehydrocholic acid conforms to the Beer - Lambert law for 0·05 to 0·1 mg of the acid per ml, the fluorescence produced by Liebermann's reagent can be used for concn. between 0·08 and 0·60 mg per ml and the absorption of the yellow-orange solution produced by the action of Straub's reagent for concn. between 1 and 10 mg per ml. The 2 : 4-dinitrophenylhydrazone, which melts at 260° C, has a max. absorption at 360 mμ. R. C. MURRAY

3064. **The quantitative determination of fructose in human semen.** J. Raboch and J. Hradec (*Endokrinologie*, 1954, **31** [3–4], 171–184).—One hundred and eighty one specimens of human semen were analysed photocolorimetrically for fructose by the Roe modification of the Selivanoff reaction but without deproteinisation. The specimen is kept in a calibrated vessel for 15 min. in a thermostat at 60° C, when the movement of the spermatozoa ceases. After thorough mixing, 0·1 ml is measured and diluted to 2·0 ml with distilled water. Resorcinol (2·0 ml of 0·1 per cent. soln. in 96 per cent. ethanol, and conc. HCl (6·0 ml) are added. The mixture is heated for 7 to 10 min. at 80° ± 0·5° C, cooled, and the colour is measured on a Klett - Summerson photocolorimeter against distilled water through a green No. 55 filter. Blank determinations are carried out in the same way without the semen. A 0·2 per cent. soln. of fructose is used for calibration. C. E. SEARLE

3065. **Evaluation of occult-blood tests on faeces.** J. W. B. Forshaw and G. W. Mason (*Lancet*, 1954, **ii** [10], 470–473).—The sensitivity and the effects of normal diet and medicinal iron upon the amidopyrin, benzidine, Gregersen, and guaiacum tests have been investigated. None of the tests was found to be very sensitive, all having a similar order of sensitivity on faeces, although on serial dilutions of blood the Gregersen test was much the most sensitive. A minimum of 8 to 10 ml of blood (11·8 to 14·8 g of haemoglobin per 100 ml) had to be consumed to produce a definite positive reaction with each of the tests; haemorrhage into the intestines sufficient to produce positive reactions may be several times as great in severely anaemic patients as in patients with normal haemoglobin. Medicinal iron *in vitro* and in faeces gave no positive reactions and restriction is unnecessary.
H. F. W. KIRKPATRICK

3066. **New solvent system for separation of amino-acids by paper chromatography.** R. A. Clayton and F. M. Strong (*Anal. Chem.*, 1954, **26** [8], 1362–1363).—The movements of 24 amino-acids have been studied with the solvent system ethyl methyl ketone, propionic acid and water (75:25:30 by vol.). R_F differences among the individual amino-acids are as great as those reported with any of the existing uni-directional systems. Duplicate R_F values agreed within $\pm$ 2 per cent. Spots were detected with a 0·125 per cent. soln. of ninhydrin in butanol. R_F values of the 24 amino-acids are listed. G. P. Cook

3067. **Paper-chromatographic analysis of [amino] acids (horizontal migration method). III.** V. K. Mohan Rao (*J. Sci. Ind. Res., B, India*, 1954, **13** [5], 342–345).—The R_F values of amino-acids have been determined in solvents containing volatile organic acids and phenol, and the influence of such factors as the distance of the spot from the solvent source and the time of irrigation have been investigated. Discrepancies in the circular R_F values of amino-acids obtained by earlier workers are shown to be due to differences in technique. The relationship between the R_M values and the molar fraction of water in the non-aqueous phase was linear for the amino-acids studied. G. C. Jones

3068. **Microchromatography. I. A technique for separation and identification of amino-acids, sugars, etc.** K. Lakshminarayanan (*Arch. Biochem. Biophys.*, 1954, **51** [2], 367–370).—The technique previously reported (*Anal. Abstr.*, 1954, **1**, 2037) has been adapted, and a microscopic method of examination of the chromatograms for accurate measurement of the R_F values of amino-acids and sugars in quantities of the order of 0·25 μg is described. The chromatograms are developed by known methods, mounted on microscope slides under cover slips and examined under the microscope with suitable colour filters. Consistent and reproducible R_F values comparable to those of larger chromatograms have been recorded N. E.

3069. **Utilisation of ion-exchangers for the separation of some amino-acids formed in the course of enzymatic degradation of cysteinesulphinic acid. Application to the isolation of hypotaurine (2-aminoethanesulphinic acid).** B. Bergeret and F. Chatagner (*Biochim. Biophys. Acta*, 1954, **14** [4], 543–550).—Mixtures of amino-acids resulting from the metabolic breakdown of cysteinesulphinic acid can be separated by the following scheme. The mixture is passed through an alumina column and the filtrate contains taurine, hypotaurine and alanine. These acids can be separated on Permutit 50, taurine occurring in the filtrate, hypotaurine being eluted by 0·002 *N* HCl and alanine by aq. NH_3. The eluate from the alumina column can be separated on Permutit 50 also, the filtrate containing cysteic acid and cysteinesulphinic acid. The dicarboxylic acids are retained, these can be later eluted with aq. NH_3. Cysteic acid and cysteinesulphinic acid can be separated on Amberlite IRA 400, cysteinesulphinic acid being eluted with 2 *N* acetic acid and cysteic acid with 0·1 *N* HCl. G. W. Cambridge

3070. **Analytical chemistry of the proteins and amino-acids.** H. D. Springall (Royal Institute of Chemistry Monograph, No. 4, 1954, 17 pp.).—This lecture (with 37 references together with a short bibliography of monographs and reviews on protein structural chemistry) deals with the determination of the amino-acids of frequent occurrence in proteins, methods for the separation of amino-acids from protein hydrolysates, and the determination of amino-acid residue sequence. The various methods and techniques are illustrated by reference to insulin. J. N. Ashley

3071. **The titration curve of gelatin.** A. W. Kenchington and A. G. Ward (*Biochem. J.*, 1954, **58** [2], 202–207).—A rapid method for determination of the titration curve of gelatin is described, and the accuracy is discussed. Revised criteria are given for the interpretation of the titration curve. The anomalous values for the histidine content of gelatin obtained by titration-curve analysis are brought into line with those obtained by other methods. All the analytical data obtained by use of the titration curve and isoelectric point agree with those from chromatographic analysis. J. N. Ashley

3072. **Hydrolysis of polysaccharides by a cation-exchange resin and identification of monosaccharide components by paper chromatography.** R. E. Glegg and D. Eidinger (*Anal. Chem.*, 1954, **26** [8], 1365–1367).—The analytical aspects of this technique are described, including control experiments to test the effect of hydrolysis medium on the various classes of monosaccharides and on di-, tri- and polysaccharides. Treated and untreated solutions were analysed quant. by chromatography and the comparison of results yielded information as to the degree of decomposition of the monosaccharides and the degree of hydrolysis of the di-, tri-, and polysaccharides under various conditions. Suitable conditions were determined for the identification of monosaccharides by chromatography after hydrolysis of carbohydrates or carbohydrate - protein complexes. The resin used was Permutit Q. G. P. Cook

3073. **Colorimetric estimation of sugars using benzidine.** J. K. N. Jones and J. B. Pridham (*Biochem. J.*, 1954, **58** [2], 288–290).—A colorimetric method, in which benzidine in acetic acid is used, is described for the determination of aldopentoses, methylaldopentoses, aldohexoses, hexuronic acids, methylated sugars and disaccharides. It cannot be used for determination of ketoses and amino-sugars. An orange-yellow colour is formed when the sugar is heated with the reagent containing a small amount of $SnCl_2$, and there is a linear relation between absorption and concn. The absorption is determined photo-electrically with the aid of an Ilford 601 filter. The error is $< \pm 3$ per cent. The method is applicable to 20 to 600 μg per ml of aldopentose, methylaldopentose, and aldohexose, 50 to 600 μg per ml of hexuronic acids, and not less than 100 μg per ml of methylated sugars and disaccharides. Polysaccharides, *e.g.*, lucerne seed galactomannan and *Lilium umbellatum* glucomannan, are first hydrolysed and the constituent sugars are separated on paper chromatograms. The sugars are then eluted from the paper strips by water, and are then determined by the benzidine method. The following give no coloration with the reagent: glycine, tryptophan, tyrosine, asparagine, cystine, hydroxyproline, urea, hexitols, and Ca gluconate. [See also *Anal. Abstr.*, 1954, **1**, 311] J. N. Ashley

3074. **Quantitative colorimetric assay of acid mucopolysaccharides.** N. DiFerrante (*J. Biol. Chem.*, 1954, **209** [2], 579–583).—The action of acid mucopolysaccharides (chondroitin sulphate and hyaluronate) in binding Fe^{III} and thereby becoming

insoluble is used as the basis for micro- and macro-methods for determination of these materials. The Fe^{III} complex is pptd. at pH 6 (0·2 *M* Na acetate - acetic acid) by aq. $FeCl_3$; the ppt. is removed by centrifugation and dissolved in dil. HNO_3. The solution is treated with aq. NH_3 and then with thioglycollic acid. The optical density of the resulting cherry-red colour is determined spectrophotometrically at 540 mμ. The amount of mucopolysaccharide is then ascertained from a standard graph. The accuracies of the micro- and macro-method are ± 2 μg and ± 10 μg, respectively. The micro-method is useful for the assay of purified preparations of hyaluronidase. The enzyme is incubated with either substrate, and the determination is then effected as described. Control and standard determinations are carried out at the same time. The method cannot be used to assay crude enzyme preparations having high protein contents because the protein interferes with pptn. of the Fe^{III} - polysaccharide complex. J. N. ASHLEY

3075. **Pentose phosphates formed by muscle aldolase.** W. L. Byrne and H. A. Lardy (*Biochim. Biophys. Acta*, 1954, **14** [4], 495–501).—Details are given of the conditions required for the separation of pentose phosphates by ion-exchange chromatography by means of Dowex 1 and dilute HCl (0·025 to 0·1 *N*) for elution. The following were separated: orthophosphate, diose phosphate, triose phosphate, xylulose 1-phosphate, fructose diphosphate and xylulose diphosphate.
G. W. CAMBRIDGE

3076. **The micro-determination of copper in cerebral tissue.** C. Chatagnon and P. Chatagnon (*Bull. Soc. Chim. Biol.*, 1954, **36** [6–7], 911–920).—Copper (1 to 4 μg) in the cerebral tissues of rats, pigs or humans is liberated from protein combination by hydrolysis with 2 *N* HCl for 20 min. at room temp. The proteins are pptd. with trichloroacetic acid, and the Cu in the supernatant liquid is estimated colorimetrically with Na diethyldithiocarbamate at pH 9. Fresh or dried defatted brain is used, and the method may also be applied to the routine analysis of Cu in blood and biological fluids.
C. E. SEARLE

3077. **The light insensitivity of the Liebermann - Burchard reaction during spectrophotometric determination of cholesterol.** J. J. Kabara (*J. Lab. Clin. Med.*, 1954, **44** [2], 246–249).—Exposure to light during colour formation is shown to affect the final colour between 340 and 540 mμ, but not between 580 and 740 mμ, in either polar or non-polar solvents. If the absorption is measured at 625 mμ (max.) no special precautions to exclude light are necessary.
W. H. C. SHAW

3078. **The measurement of phosphorus-32 labelling of individual kephalins and lecithin in a small sample of tissue.** R. M. C. Dawson (*Biochim. Biophys. Acta*, 1954, **14** [3], 374–379).—A method is reported for measurement of the ^{32}P-labelling of phosphatidyl ethanolamine, phosphatidyl serine, phosphatidyl choline and diphosphoinositide in a single small sample (150 to 400 mg) of tissue. The lipids are isolated from the tissue and hydrolysed by methanolic KOH; after removal of alkali and fatty acids the breakdown products of the phospholipids are resolved by two-dimensional chromatography on filter-paper. Details are given of these procedures. The spots are located by spraying with the acid - molybdate reagent of Hanes and Isherwood (*Brit. Abstr. C*, 1950, 402). The phosphorus contained in the spots is dissolved by the technique of Ansell and Dawson (*Biochem. J.*, 1951, **50**, 241) and the radioactivity is measured in a liquid-counter tube. G. W. CAMBRIDGE

3079. **Hydroxyproline as an index of connective tissue in muscle.** E. Wierbicki and F. E. Deatherage (*J. Agric. Food Chem.*, 1954, **2** [17], 878–882).—The method of Neuman and Logan (*Brit. Abstr. C*, 1950, 412) was adapted to this determination by the use of alkaline or acid hydrolysates. Acid hydrolysis of muscle tissue in presence of $SnCl_2$ was superior to alkaline hydrolysis. The material used was the *longissimus dorsi* muscle of cattle. The sample was treated with 12 *N* HCl and 0·7 g of $SnCl_2$ (approx. three-quarters of the wt. of protein) and refluxed for 7 hr. The hydrolysate was neutralised and filtered. The apparent absorbance at 560 mμ was corrected for tyrosine by subtracting 0·072 absorbance unit and an additional correction of 0·010 absorbance unit for the yellow colour of the hydrolysate. A method for the determination of collagen and elastin in connective tissue is suggested. *Longissimus dorsi* muscles contained 12·39 ± 0·40 per cent. of hydroxyproline and consisted of 84 per cent. of collagen and 16 per cent. of elastin. Fat tended to destroy tryptophan during alkaline hydrolysis of tissue. The tyrosine and tryptophan contents of *longissimus dorsi* muscle were 1·024 ± 0·047 per cent. and 0·330 ± 0·020 per cent., respectively. S.C.I. ABSTR.

3080. **Estimation of pentose nucleic acid and deoxypentose nucleic acid in the liver and brain tissue of mice following the feeding of the insecticide Aldrin (hexachlorohexahydrodimethanonaphthalene).** E. Annau (*Canad. J. Biochem. Physiol.*, 1954, **32** [3], 178–183).—Mouse liver tissue (approx. 1 g) was ground with sand and suspended in ice-cold 7 per cent. trichloroacetic acid. After stirring the mixture for 15 min., an equal vol. of pure ethanol was added. The mixture was stirred at 0° C for a further 5 min. and filtered. The residue was washed with ice-cold 1 per cent. trichloroacetic acid, water and 2 per cent. $NaHCO_3$, and then dried with pure ethanol and ether. The residue was finely ground and extracted twice with 20 to 30 ml of a (3 + 1) mixture of ethanol and ether by boiling and centrifuging, and then with 50 ml of a (1 + 1) mixture of methanol and $CHCl_3$ by boiling for 30 min., filtering and washing with ether. The air-dried residue was suspended in 0·5 *N* KOH, incubated for 18 hr. at 38° C and centrifuged. Acid fractionation and chemical analyses were carried out on the supernatant liquid as indicated by the method of Schmidt and Thannhauser (*J. Biol. Chem.*, 1945, **161**, 83). The method of Delory (*Biochem. J.*, 1938, **32**, 5) was used for pptn. of inorganic P, which was determined by the method of Fiske and Subbarow (*J. Biol. Chem.*, 1925, **66**, 375). Mouse brains (two) were homogenised at 0° C with 4 ml of H_2O, 6 ml of acetone (at 0° C) were added and the mixture was stirred for 15 min. After centrifuging, the sediment was washed with a (3 + 2) mixture of acetone and H_2O and then with pure acetone, suspended in a (3 + 1) mixture of ethanol and ether, boiled for 5 min. and centrifuged. The sediment was extracted for 20 min. with hot (1 + 1) methanol - $CHCl_3$ and finally washed with ether. The air-dried residue was suspended in 20 ml of *N* KOH, stirred for 45 min. and then incubated at 38° C for 18 hr. After centrifuging, the clear yellow supernatant liquid was fractionated and analysed as above.
H. F. W. KIRKPATRICK

3081. **Formation of urocanic acid and glutamic acid in the fermentation of histidine by *Clostridium tetanomorphum*. [Determination of urocanic acid.]** R. L. Wickremasinghe and B. A. Fry (*Biochem. J.*, 1954, **58** [2], 268–278).—A u.v. spectrophotometric method for determination of urocanic acid [β-glyoxalin-4(or 5)-ylacrylic acid] is described. This acid has a characteristic u.v. absorption spectrum, and, although the position of the peak (≃ 276 mμ at pH 7) depends on pH, the $E^{1\%}_{1\,cm}$ max. value varies little in the pH range of 4 to 10. The spectrum is constant for 24 to 48 hr., but when possible the assay should be carried out within 24 hr. The pH is adjusted to a pre-determined value by adding a buffer, *e.g.*, an equal vol. of 0·2 *M* Sörensen phosphate buffer, pH 7·2. Under these conditions there is a linear relationship between extinction at 276 mμ and concn. in the range of 1 to 10 μg per ml.
J. N. ASHLEY

3082. **Aldosterone. Isolation and properties. The constituents of the suprarenal gland and related substances.** S. A. Simpson, J. F. Tait, A. Wettstein, R. Neher, J. v. Euw, O. Schindler and T. Reichstein (*Helv. Chim. Acta*, 1954, **37** [4], 1163–1200).—The preparation, structure and properties of aldosterone (**I**) are described. By double paper chromatography a semiquantitative assay of **I** in blood or suprarenal glands is possible. A preliminary chromatographic separation on silica gel may be necessary. The residue, after an appropriate extraction procedure) from suprarenals or blood, is transferred to a Whatman No. 1 filter-paper and chromatographed for 2½ hours with a toluene - ethyl acetate - methanol - water mixture (45:5:25:25). **I** is detected by an u.v. (253 mμ) photographic technique and the strip containing it is carefully cut out and eluted with methanol - water (1 + 5); **I** is extracted from this eluate with chloroform - ether (1 + 3). The extract is spotted on paper and chromatographed with a propylene glycol - toluene mixture for 20 hr. The paper is then dried and passed through a solution containing 0·01 per cent. tetrazolium blue in 0·2 *N* NaOH. The blue colour that immediately develops is compared visually with standard cortisone spots similarly obtained.
P. S. STROSS

3083. **Factors affecting the bio-assay of 11-oxycorticosteroids in adrenalectomised rats.** N. R. Stephenson (*Canad. J. Biochem. Physiol.*, 1954, **32** [3], 218–226).—Determination of total sugar by copper reduction in deproteinised liver hydrolysates was found to be an acceptable criterion of liver glycogen deposition response in the bio-assay of 11-oxycorticosteroids. Total carbohydrate determined by anthrone in liver hydrolysates was also shown to be suitable. The liver was removed, blotted on filter-paper, weighed and dropped into 10 ml of *N* H_2SO_4 soln. The soln. was heated in a bath of boiling water for 30 min., the liver being broken up by a glass rod, and then heated in an autoclave at 15 lb pressure for 15 to 20 min. After cooling, the soln. was neutralised and made up to 30 ml with water. A 2-ml portion was deproteinised with $Zn(OH)_2$ and the glucose equivalent of protein-free hydrolysate was determined by the method of Nelson (*J. Biol. Chem.*, 1944, **153**, 375). A 5-ml aliquot of the hydrolysate was kept cold while mixing it with 10 ml of anthrone reagent (0·15 per cent. in 95 per cent. v/v H_2SO_4), and the mixture was heated for 10 min. in boiling water. The resulting colour was read at 625 mμ. Glucose was used as reference standard in both procedures. In order to keep the mortality rate as small as possible, rats were adrenalectomised at 9 a.m., starved overnight and used in the assay the following day. This procedure does not influence the precision unfavourably. Glucose administration increases glycogen deposition but ethanol appears to depress it, a lower response being attained with cortisone in ethanol soln. than with the same steroid in oil soln.
H. F. W. KIRKPATRICK

3084. **The assay of thrombin.** D. M. Bryce (*Pharm. J.*, 1954, **172**, 532–534).—The units of the British Pharmacopoeia and U.S. National Institute of Health are compared and discussed. The B.P. and N.I.H. assay methods are unnecessarily crude, as thrombin can be assayed by the normal "measured effect" bio-assay by application of the rectilinear relation between log-concn. and log- (clotting time). Assay methods and the characteristics of various substrates are discussed.
S. C. JOLLY

3085. **A photo-electric method for the rapid determination of moisture in biological materials.** G. S. Siddappa and D. P. Das (*Curr. Sci.*, 1954, **23** [5], 157–158).—A modification of the method of Launer and Tomimatsu (*Brit. Abstr. C*, 1952, 516), is described. The organic substance in the material is oxidised with dichromate and sulphuric acid, and the resulting yellowish green colour is measured in a photo-electric absorptiometer with a 660-mμ filter. The colour is related to the moisture content of a particular type of material by means of a standard reference curve derived from results obtained on samples containing known amounts of completely dried material. Results obtained on mango and papaya pulp at different stages of the dehydration process show agreement within 2·6 per cent. with those obtained by drying the samples for 55° C for 72 hr.; the error is low.
E. HAYES

See also Abstracts 3103, 3108.

Drugs

3086. **Symposium on the comparison of chemical and biological estimation of drugs in quantitative pharmacology.** (*Analyst*, 1954, **79**, 511–518).—Six papers presented and discussed at a meeting of The Society for Analytical Chemistry are summarised. The subjects were: chemical methods for determination of digitalis; the biological standardisation of digitalis; chemical determination of vitamin D; biological methods of assay of vitamin D; chemical and biological methods for the determination of adrenaline and noradrenaline; routine methods used in the quant. determination of adrenaline.
A. O. JONES

3087. **Comparative studies of methods of evaluating antibacterial substances. I. Evaluation of bacteriostatic action.** A. M. Cook (*J. Pharm. Pharmacol.*, 1954, **6** [9], 629–637).—Three methods of measuring bacteriostasis, *viz.*, incubation of the bacteriostatic agent with liquid culture medium inoculated with the test organism, incorporation of the agent in a solid medium seeded with the test organism, and a counting method, have been examined. A fair degree of correlation was found between the first and second methods. The third supports Price's hypothesis (*Ann. N.Y. Acad. Sci.*, 1950, **53**, 84) that there is no real difference between bacteriostatic and bactericidal actions.
S. C. JOLLY

3088. **Comparative studies of methods of evaluating antibacterial substances. II. Evaluation of bactericidal action. A comparison of an extinction**

method with a counting method. A. M. Cook and B. A. Wills (*J. Pharm. Pharmacol.*, 1954, **6** [9], 638–648).—The bactericidal action of phenol on *E. coli* examined by the method of Berry and Bean (*Anal. Abstr.*, 1954, **1**, 3089) is compared with that given by a counting technique. There is correlation between the mean single survivor time and the percentage of survivors at a series of shorter contact times as estimated by serial dilution counts.
S. C. JOLLY

3089. **The estimation of bactericidal activity from extinction time data.** H. Berry and H. S. Bean (*J. Pharm. Pharmacol.*, 1954, **6** [9], 649–655).—A new method is described for estimating extinction times with a reproducibility within limits sufficiently close to invalidate other extinction time methods. The method involves sampling the reaction mixture immediately after mixing the organisms with the bactericide, and transferring the sample to sterile tubes maintained at constant temp., when the reaction proceeds until quenched by addition of sufficient sterile broth to inactivate the bactericide. The method is performed in replicate, a dropping pipette instead of the usual loop is used for sampling the reaction mixture, and relatively narrow limits ($\frac{1}{5}$ to $\frac{1}{10}$ of the anticipated death times) are used for the exposure time intervals. The method is illustrated by the disinfection of *E. coli* by aqueous solutions of phenol, and is applicable to systems containing water-soluble phenols solubilised in soap solutions where clumping might invalidate other methods.
S. C. JOLLY

3090. **Paper chromatography of alkaloids with solutions of electrolytes.** A. Resplandy (*Compt. Rend.*, 1954, **238** [26], 2527–2529).—Optimum conditions for systematic detection and identification of certain alkaloids by ascending paper chromatography with electrolyte solutions only are investigated. R_F values are independent of the amount of alkaloid (10 to 50 μg) in soln., and are the same for both salts and bases. The chromatograms should be developed in an atmosphere having a R.H. $\simeq$ 100 per cent.; although R_F values change with temp., variations of $\pm$ 3° C are permissible. The effect of solution pH and of different anions and cations on R_F values is indicated briefly; $(NH_4)_2SO_4$ in concn. of 200 to 500 g per litre is the preferred electrolyte, especially for the clear separation of two or more alkaloids having close R_F values, *e.g.*, tropine, ephedrine, atropine and diamorphine. Once optimum concn. and pH of electrolyte giving clearly-separated distinct spots are determined, identification is effected by reference to a standard series of R_F values (*cf.* Munier, *Brit. Abstr. C*, 1952, 163).
W. J. BAKER

3091. **Chromatography on paper: preliminary tests with mixtures of alkaloids.** G. Thomas and P. Roland (*Ann. Pharm. Franç.*, 1954, **12** [4], 318–320).—Paper chromatography of a number of alkaloids of pharmaceutical importance is carried out with butanol saturated with water on Whatman No. 1 paper buffered by dipping in 0·2 *M* KH_2PO_4. The ratio of distance travelled by the compound to distance travelled by morphine is found a more reproducible measure than the R_F for identification.
E. J. H. BIRCH

3092. **Chromatographic separation on paper of some alkaloids as their reineckates.** M. Milletti and G. Adembri (*Sperimentale*, 1954, **4** [5–6], 99–104).—A technique is described for the separation of various alkaloids by the paper chromatography of solutions of their reineckates in acetone (0·05 g in 10 ml) by means of pyridine - water mixtures (1 + 4) as eluting soln. Mean R_F values are reported for chromatography on discs and strips of paper for 17 alkaloids.
R. C. MURRAY

3093. **A specific method for the determination of morphine.** R. R. A. Pride and E. S. Stern (*J. Pharm. Pharmacol.*, 1954, **6** [9], 590–606).—A routine method is described for the determination of morphine based on the iodic acid - ammonium carbonate - nickel salt reaction. The solution of morphine is first adjusted to pH 4 to 5, and then diluted until the morphine concn. (approx. 15 to 30 mg per 100 ml) is such that extinction readings are within the optimum range for the instrument; during dilution the HCl concn. is adjusted to 0·05 *N* (pH $<$ 1·6). A 10-ml aliquot is treated for exactly 2 min. with 2 ml of 4·5 per cent. w/v iodic acid soln., then 10 ml of complexing reagent (8·0 g of NH_4HCO_3 shaken with 25 ml of 21·4 per cent w/v NH_4Cl soln., 20 ml of 1·0 *N* aq. NH_3 and 10 ml of 1·0 per cent w/v $NiCl_2.6H_2O$ soln., and diluted to 100 ml, prepared fresh each day) is added, the solution is diluted to 25 ml, and the extinctions at 530 and 670 mμ are determined after 90 min. The pH of the solution at the end of this period should be 8·00 $\pm$ 0·05. A blank determination is carried out with 5 ml of 0·1 *N* HCl in place of the iodic acid soln. The morphine content is determined by reference to a calibration curve at 670 mμ prepared from solutions containing 4·0 to 40·0 mg of anhydrous morphine per 100 ml, and corrected, if required, by application of a factor for "pseudomorphine-like" background colour. Suitable methods of extracting morphine from opium and poppy capsules are described. The method is sensitive to as little as 0·005 per cent. of morphine, and 0·3 per cent. can be determined with an accuracy within $\pm$ 2 per cent. Of the bases occurring naturally with morphine, only pseudomorphine interferes.
S. C. JOLLY

3094. **Paper chromatography applied to the detection of opium alkaloids in urine and tissues.** G. J. Mannering, A. C. Dixon, N. V. Carroll and O. B. Cope (*J. Lab. Clin. Med.*, 1954, **44** [2], 292–300).—R_F values are given for seven opium alkaloids and for ethylmorphine, diamorphine and meperidine (Demerol) at different pH values and with different solvent mixtures. Procedures are detailed for preparing extracts of urine and tissues for chromatography; spray reagents for the detection of the substances are also described. After extraction from the completed chromatograms, the purified substances are identified by conventional colour reagents. Minute amounts of morphine are detectable by the method.
W. H. C. SHAW

3095. **The determination of small quantities of atropine in pharmaceutical mixtures.** M. I. Kuleshova (*Aptechnoe Delo*, 1954, **3** [1], 13–17).—Tungstophosphoric acid gives a detectable cloudiness with a soln. containing 0·0091 mg per ml of atropine in 1 per cent. HCl. The atropine is extracted and dissolved in 1 per cent. HCl, the soln. is diluted to the limiting concn. (based on the expected atropine content) and tungstophosphoric acid is added; the cloudiness is compared with standards treated in the same way. If necessary, the test soln. is diluted so that the cloudiness is the same as that given by a standard of the limiting concn., and the atropine content is calculated from the degree of dilution. The determination should be carried out between 15° and 22° C.
E. HAYES

3096. **Melting points with decomposition and the heat stability of atropine sulphate.** E. I. Rosenblum and W. S. Taylor (*J. Pharm. Pharmacol.*, 1954, **6** [9], 656–659).—Decomposition m.p. are affected chiefly by rate and time of heating. The results by the B.P. method for determining the m.p. of atropine sulphate were more concordant than were those obtained by the U.S.P. method, but even with the B.P. method considerable replication is necessary to obtain a reliable figure. Sulphates of several ester-alkaloids are apparently more heat sensitive than are the hydrobromides and hydrochlorides. The "heat stability" of decomposable substances may give a useful indication of purity.
S. C. JOLLY

3097. **Identification of cardiac glycosides and aglycones in strophanthus seeds by paper chromatography.** E. Heftmann, P. Berner, A. L. Hayden, H. K. Miller and E. Mosettig (*Arch. Biochem. Biophys.*, 1954, **51** [2], 329–339).—This is a report on the cardiac glycosides and aglycones that were found in about 100 seed samples of the genus *Strophanthus* by paper chromatography. The glycosides were extracted by the method of Schindler and Reichstein (*Brit. Abstr. C*, 1951, 464) or of Bush and Taylor (*Biochem. J.*, 1952, **52**, 643), which gave a better yield. Hydrolysis was carried out by the Mannich and Siewert method (*Ber.*, 1942, **75**, 737). The chromatographic method of Heftmann and Levant (*Brit. Abstr. C*, 1952, 499) was followed, and Tollens' reagent was used for detection. The identity of the steroids was confirmed by acetylation of the seed extracts or hydrolysis followed by acetylation.
N. E.

3098. **Determination of cardiotonic glycosides with a reagent composed of ferric chloride, sulphuric acid and glacial acetic acid.** D. H. E. Tattje (*Ann. Pharm. Franç.*, 1954, **12** [4], 267–275).—The use of $FeCl_3$ - H_2SO_4 - acetic acid as a reagent for digitoxose in glycosides is briefly reviewed. The effect of variation of the components of the reagent is investigated by use of 0·6 mg of digitoxin. The effect of variation of $FeCl_3$ is not large, but lower concn. leads to a slower development, but a longer duration of max. extinction. Increase in the amount of water stabilises the absorption of the max., which, however, is less. Increase in the amount of H_2SO_4 increases the max. and shortens the time of attainment, but also shortens the time of its persistence. Absorption curves for the reaction are shown. The recommended reagent contains 1 ml of 5 per cent. aq. $FeCl_3$, 1·5 ml of H_2O, 5 ml of H_2SO_4 and acetic acid to 100 ml; the absorption is measured at 600 mμ.
E. J. H. BIRCH

3099. **Detection of scilliroside in organic material.** F. Dybing, O. Dybing and K. B. Jensen (*Acta Pharmacol. Tox., Kbh.*, 1954, **10** [2], 93–100).—Scilliroside, the active constituent of red squill, is separated by paper chromatography and then detected biologically in mice. Ten to fifteen μg of substance can be detected in this way, or 1 to 10 μg by three (non-specific) colour tests. Methods for isolating scilliroside from biological material are detailed.
W. H. C. SHAW

3100. **Method for the determination of tannins in vegetable materials and galenicals.** S. M. Bolotnikov and M. S. Shraiber (*Aptechnoe Delo*, 1954, **3** [3], 10–14).—The following methods of determining tannins are compared: (*i*) pptn. with Cu acetate soln. by the U.S.S.R. Pharmacopoeia VIII method, (*ii*) Löwenthal's method with the use of carbon, (*iii*) Löwenthal's method without the use of carbon, (*iv*) oxidation with $K_3Fe(CN)_6$, and (*v*) the international hide-powder method. The results obtained by method (*iv*) are too high, but those obtained by the other methods are in good agreement; method (*iii*) is recommended. In this method polyphenols interfere, but they may be removed by a single one-hour maceration with ether. The most complete extraction of tannins is obtained by macerating the vegetable material, previously ground to pass a 3-mm mesh sieve, with 40 per cent. ethanol.
E. HAYES

3101. **Assay of mandelic acid and its pharmaceutical preparations by cerimetric method.** M. R. Verma and S. D. Paul (*J. Sci. Ind. Res., B, India*, 1954, **13** [5], 346–348).—Methods of estimating mandelic acid are discussed. The oxidation with ceric sulphate is used in the development of a simple and speedy method for the direct determination of mandelic acid and its salts in pharmaceutical preparations, without interference by phosphates, bicarbonates, etc., even when these are present in large quantities. The sample is boiled with dil. H_2SO_4 and ceric sulphate for 25 min. and excess of Ce^{IV} is titrated with ferrous ammonium sulphate with ferroin indicator.
G. C. JONES

3102. **The quantitative determination of barbiturates in pharmaceutical mixtures.** G. A. Vaisman and L. I. Rapoport (*Aptechnoe Delo*, 1954, **3** [1], 17–25).—For analytical purposes pharmaceutical mixtures containing barbiturates can be classified as follows. (*a*) Those containing ether-insoluble compounds such as Na salicylate, Ca lactate, Ca glycerophosphate, theobromine and sodium salicylate, papaverine hydrochloride and other alkaloidal salts; the barbiturate is extracted with ether and, after treatment with Na_2CO_3 soln., is titrated with 0·1 *N* $AgNO_3$. (*b*) Those containing acetylsalicylic acid, phenazone, codeine, caffeine, sucrose, sodium bicarbonate, phenacetin, bromocamphor or bromvaletone; these do not interfere with the direct argentimetric determination. (*c*) Those containing hexamine or amidopyrine; the barbiturate in ethanol is titrated directly against 0·1 *N* alkali with thymolphthalein as indicator. (*d*) Those containing barbiturates in the form of Na salts; these are titrated against 0·1 *N* HCl, if the other ingredients do not interfere. The results of determinations on various mixtures are given.
E. HAYES

3103. **A spectrophotometric method for determining barbituric acids in body fluids.** P. Lous (*Acta Pharmacol Tox., Kbh.*, 1954, **10** [2], 134–146).—The u.v. absorption spectrum of unsubstituted barbituric acid and the influence of pH on the chloroform extraction of this substance, barbitone, phenobarbitone and allyl*iso*propylbarbituric acid are studied. Under prescribed extraction conditions that eliminate unsubstituted barbituric acid the other three barbiturates may be determined by the differential spectrophotometric method described previously (*Brit. Abstr. C*, 1951, 140). On samples of serum and urine agreement is satisfactory between the results given by the spectrophotometric method and those given by a gravimetric method which is described in detail.
W. H. C. SHAW

3104. **Methods for the determination of phenobarbitone in polyethylene glycol 400 solutions.** J. Autian and B. F. Allen (*Drug Standards*, 1954, **22** [7–8], 164–168).—Of six methods tested the Budde

method (*ApothekerZtg.*, 1934, **49**, 295) and a non-aqueous potentiometric method proved the most satisfactory. In the Budde method, 20 ml of the phenobarbitone solution were diluted with 40 ml of water to which 10 ml of 3 per cent. w/v Na_2CO_3 soln. had been added. The solution was then titrated with 0·1 *N* $AgNO_3$ soln. until the first permanent cloudiness appeared. A beam of light was passed through the solution to aid in determining the end-point. Each ml of 0·1 *N* $AgNO_3 \equiv$ 23·233 mg of phenobarbitone. In the potentiometric method, the water was removed from the preparation on a water-bath. The solution was diluted with 50 ml of $CHCl_3$ and titrated potentiometrically with 0·1 *N* Na methoxide. N. E.

3105. **Spectrophotometric determination of *iso*nicotinic acid hydrazide [isoniazid].** D. S. Goldman (*Science*, 1954, **120**, 315–316).—The method is based upon changes in the absorption spectrum of isoniazid (**I**) in aq. soln. induced by change of pH. In strongly alkaline soln., the peak, which occurs at 298 mμ (extinction coeff. $4{\cdot}8 \times 10^6$ sq. cm per mole) disappears reversibly in acid soln. NaOH is added to an aq. soln. of **I** to give a final concn. of 0·1 *N*, and the optical density is read at 298 mμ in a spectrophotometer. The lower limit of the method is about 0·5 μg of **I** per ml, with an over-all sensitivity of 0·1 μg per ml. None of the following related compounds show the absorption peak at 298 mμ: nicotinamide, *iso*nicotinamide, *iso*nicotinic acid, 1-*iso*nicotinyl-2-*iso*propylhydrazine, 2-allyl-1-*iso*nicotinyl-2-*iso*propylhydrazine dihydrochloride and N′-methyl-*iso*nicotinic acid hydrazide.

H. F. W. Kirkpatrick

3106. **The quantitative determination of mepacrine, proguanil and Plazmotsid in tablets.** G. A. Vaisman (*Aptechnoe Delo*, 1953, **2** [6], 25–30).—The powdered tablets are treated with dil. H_2SO_4 and extracted with ether; the methylenedisalicylic acid so obtained is determined bromimetrically. Mepacrine is determined by extracting with water, evaporating the filtered extract to a small vol. and adding excess of 0·1 *N* iodine monochloride in HCl. The ICl consumed is determined by adding KI and back-titrating with $Na_2S_2O_3$. Plazmotsid [the methylenedisalicylic acid salt of 8-(3-diethylaminopropylamino)-6-methoxyquinoline] does not react with ICl under the conditions described. The sum of the proguanil plus mepacrine is determined by evaporating a portion of the aqueous extract, adding ether and titrating with 0·1 *N* NaOH until the aqueous layer is alkaline to phenolphthalein. The results of determinations on various tablets and mixtures are given. E. Hayes

3107. **A contribution to the photometric estimation of acetanilide Lactophenin, phenacetin, Rodilone, Tibatin and Tebethion [thiacetazone] in pharmaceuticals.** J. Richter (*Z. anal. Chem.*, 1954, **142** [4], 277–279).—A rapid method for the colorimetric estimation of the above chemicals in pharmaceuticals is described. One to one hundred mg of any of these is hydrolysed for 5 min. with 12·5 per cent. HCl (20 ml), diluted, nearly neutralised with 15 per cent. NaOH (20 ml) or KOH (25 ml) and diazotised with 0·5 per cent. $NaNO_2$ soln. (5 ml); 0·1 per cent. thymol soln. in 1·5 per cent. NaOH (5 ml) is added, and the orange colour is measured after 2 min. Pyrazolone derivatives and salicylic acid compounds do not interfere. P. S. Stross

3108. **A new technique for performing the Cronheim - Ware reaction.** K. F. Körner (*Z. anal. Chem.*, 1954, **142** [4], 267–275).—In this critical examination of the Cronheim - Ware reaction (*J. Pharmacol. Exp. Ther.*, 1948, **92**, 98) several important modifications are suggested by which the accuracy and reproducibility are much increased. Quartz apparatus suitably treated is found to be essential, and it is found necessary to maintain constant temperature to prevent precipitation of water from the solvent layer; the buffer concentrations have also been changed. The modified method is applied to urine analysis; it was found to be applicable to the determination of strychnine, Pantocain [amethocaine], Dromoran [methorphinan], Cliradon [4-*m*-hydroxyphenyl-1-methyl-4-propionylpiperidine hydrochloride], whilst Cardiazol [leptazol], caffeine, Coramine [nikethamide], Sympatol [1-(*p*-hydroxyphenyl)-2-methylaminoethanol tartrate], Suprifen [1-(*p*-hydroxyphenyl)-2-methylaminopropanol], morphine alkaloids and many pyrazolone derivatives give negative results. P. S. Stross

3109. **The quantitative determination of Atoxyl [sodium *p*-aminophenylarsonate] by a bromimetric method.** V. A. Gvozdik, M. A. Trotsenko and V. P. Potapova (*Aptechnoe Delo*, 1954, **3** [3], 33–35).—To 10 ml of an aqueous soln. containing about 0·1 g of Atoxyl in a ground-glass flask, add 25 ml of 0·1 *N* $KBrO_3$ soln., 0·5 g of KBr and sufficient conc. HCl to make the final concn. 2 *N*; set aside the stoppered flask in the dark with frequent shaking. Add 20 ml of 0·1 *N* As_2O_3 solution, heat on a water-bath to 60° to 70° C and titrate with 0·1 *N* $KBrO_3$ (indicator, methyl orange). One ml of 0·1 *N* $KBrO_3 \equiv$ 0·005485 g of Atoxyl pentahydrate. The method gives results in agreement with those obtained by the U.S.S.R. Pharmacopoeia VIII method; the limits of error are ± 0·3 per cent. Iodimetric determination of the excess of Br in the first stage gave results less satisfactory than those obtained with the use of As_2O_3. E. Hayes

3110. **The use of flotation in testing pharmaceutical preparations for salts of heavy metals, iron and zinc.** A. G. Belousova (*Aptechnoe Delo*, 1954, **3** [3], 23–27).—The sulphide limit test for lead in pharmaceutical preparations is made more sensitive and reproducible by the addition of a mixture of *iso*butanol and ether (2 + 100) after the sulphide has been precipitated; the ppt. then forms a film at the phase boundary, where it can be more easily observed and compared with a standard. In the $K_4Fe(CN)_6$ test for iron a mixture of 100 vol. of ether, 5 vol. of ethanol and 0·75 vol. of vegetable oil is used to form the non-aqueous phase, and a 12-fold increase in sensitivity is achieved. In the $K_4Fe(CN)_6$ test for zinc, a mixture of *iso*butanol and ether (2 + 100) is used. Semi-micro adaptations of the tests are described. E. Hayes

3111. **The routine determination of the antacid efficiency of aluminium hydroxide gels.** P. R. Clemow and J. W. Lowry (*J. Pharm. Pharmacol.*, 1954, **6** [9], 610–618).—*In vitro* methods for assessing the therapeutic value of antacids are discussed in relation to the routine evaluation of aluminium hydroxide gels. A suitable routine test is as follows. Add quickly 8 g of liquid gel or 0·6 g of dried gel (or an equivalent amount if in any other form) to 250 ml of 0·05 *N* HCl at 37° ± 0·5° C (pH 1·35 to 1·40) in a 250-ml beaker. Agitate the beaker briskly and continuously and determine the pH at intervals. It is suggested that with the dried gel, a pH of ≮ 3·0 should be attained in 20 min. and a pH of ≮ 3·5 in 30 min., and at no time should

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3098. **Determination of cardiotonic glycosides with a reagent composed of ferric chloride, sulphuric acid and glacial acetic acid.** D. H. E. Tattje (*Ann. Pharm. Franç.*, 1954, **12** [4], 267–275).—The use of $FeCl_3$ - H_2SO_4 - acetic acid as a reagent for digitoxose in glycosides is briefly reviewed. The effect of variation of the components of the reagent is investigated by use of 0·6 mg of digitoxin. The effect of variation of $FeCl_3$ is not large, but lower concn. leads to a slower development, but a longer duration of max. extinction. Increase in the amount of water stabilises the absorption of the max., which, however, is less. Increase in the amount of H_2SO_4 increases the max. and shortens the time of attainment, but also shortens the time of its persistence. Absorption curves for the reaction are shown. The recommended reagent contains 1 ml of 5 per cent. aq. $FeCl_3$, 1·5 ml of H_2O, 5 ml of H_2SO_4 and acetic acid to 100 ml; the absorption is measured at 600 mμ.
E. J. H. BIRCH

3099. **Detection of scilliroside in organic material.** F. Dybing, O. Dybing and K. B. Jensen (*Acta Pharmacol. Tox., Kbh.*, 1954, **10** [2], 93–100).—Scilliroside, the active constituent of red squill, is separated by paper chromatography and then detected biologically in mice. Ten to fifteen μg of substance can be detected in this way, or 1 to 10 μg by three (non-specific) colour tests. Methods for isolating scilliroside from biological material are detailed.
W. H. C. SHAW

3100. **Method for the determination of tannins in vegetable materials and galenicals.** S. M. Bolotnikov and M. S. Shraiber (*Aptechnoe Delo*, 1954, **3** [3], 10–14).—The following methods of determining tannins are compared: (*i*) pptn. with Cu acetate soln. by the U.S.S.R. Pharmacopoeia VIII method, (*ii*) Löwenthal's method with the use of carbon, (*iii*) Löwenthal's method without the use of carbon, (*iv*) oxidation with $K_3Fe(CN)_6$, and (*v*) the international hide-powder method. The results obtained by method (*iv*) are too high, but those obtained by the other methods are in good agreement; method (*iii*) is recommended. In this method polyphenols interfere, but they may be removed by a single one-hour maceration with ether. The most complete extraction of tannins is obtained by macerating the vegetable material, previously ground to pass a 3-mm mesh sieve, with 40 per cent. ethanol.
E. HAYES

3101. **Assay of mandelic acid and its pharmaceutical preparations by cerimetric method.** M. R. Verma and S. D. Paul (*J. Sci. Ind. Res., B, India*, 1954, **13** [5], 346–348).—Methods of estimating mandelic acid are discussed. The oxidation with ceric sulphate is used in the development of a simple and speedy method for the direct determination of mandelic acid and its salts in pharmaceutical preparations, without interference by phosphates, bicarbonates, etc., even when these are present in large quantities. The sample is boiled with dil. H_2SO_4 and ceric sulphate for 25 min. and excess of Ce^{IV} is titrated with ferrous ammonium sulphate with ferroin indicator.
G. C. JONES

3102. **The quantitative determination of barbiturates in pharmaceutical mixtures.** G. A. Vaisman and L. I. Rapoport (*Aptechnoe Delo*, 1954, **3** [1], 17–25).—For analytical purposes pharmaceutical mixtures containing barbiturates can be classified as follows. (*a*) Those containing ether-insoluble compounds such as Na salicylate, Ca lactate, Ca glycerophosphate, theobromine and sodium salicylate, papaverine hydrochloride and other alkaloidal salts; the barbiturate is extracted with ether and, after treatment with Na_2CO_3 soln., is titrated with 0·1 *N* $AgNO_3$. (*b*) Those containing acetylsalicylic acid, phenazone, codeine, caffeine, sucrose, sodium bicarbonate, phenacetin, bromocamphor or bromvaletone; these do not interfere with the direct argentimetric determination. (*c*) Those containing hexamine or amidopyrine; the barbiturate in ethanol is titrated directly against 0·1 *N* alkali with thymolphthalein as indicator. (*d*) Those containing barbiturates in the form of Na salts; these are titrated against 0·1 *N* HCl, if the other ingredients do not interfere. The results of determinations on various mixtures are given.
E. HAYES

3103. **A spectrophotometric method for determining barbituric acids in body fluids.** P. Lous (*Acta Pharmacol Tox., Kbh.*, 1954, **10** [2], 134–146).—The u.v. absorption spectrum of unsubstituted barbituric acid and the influence of pH on the chloroform extraction of this substance, barbitone, phenobarbitone and allyl*iso*propylbarbituric acid are studied. Under prescribed extraction conditions that eliminate unsubstituted barbituric acid the other three barbiturates may be determined by the differential spectrophotometric method described previously (*Brit. Abstr. C*, 1951, 140). On samples of serum and urine agreement is satisfactory between the results given by the spectrophotometric method and those given by a gravimetric method which is described in detail.
W. H. C. SHAW

3104. **Methods for the determination of phenobarbitone in polyethylene glycol 400 solutions.** J. Autian and B. F. Allen (*Drug Standards*, 1954, **22** [7–8], 164–168).—Of six methods tested the Budde

method (*ApothekerZtg.*, 1934, **49**, 295) and a non-aqueous potentiometric method proved the most satisfactory. In the Budde method, 20 ml of the phenobarbitone solution were diluted with 40 ml of water to which 10 ml of **3** per cent. w/v Na_2CO_3 soln. had been added. The solution was then titrated with 0·1 *N* $AgNO_3$ soln. until the first permanent cloudiness appeared. A beam of light was passed through the solution to aid in determining the end-point. Each ml of 0·1 *N* $AgNO_3 \equiv$ 23·233 mg of phenobarbitone. In the potentiometric method, the water was removed from the preparation on a water-bath. The solution was diluted with 50 ml of $CHCl_3$ and titrated potentiometrically with 0·1 *N* Na methoxide. N. E.

3105. **Spectrophotometric determination of *iso*nicotinic acid hydrazide [isoniazid].** D. S. Goldman (*Science*, 1954, **120**, 315–316).—The method is based upon changes in the absorption spectrum of isoniazid (**I**) in aq. soln. induced by change of pH. In strongly alkaline soln., the peak, which occurs at 298 mμ (extinction coeff. $4{\cdot}8 \times 10^6$ sq. cm per mole) disappears reversibly in acid soln. NaOH is added to an aq. soln. of **I** to give a final concn. of 0·1 *N*, and the optical density is read at 298 mμ in a spectrophotometer. The lower limit of the method is about 0·5 μg of **I** per ml, with an over-all sensitivity of 0·1 μg per ml. None of the following related compounds show the absorption peak at 298 mμ: nicotinamide, *iso*nicotinamide, *iso*nicotinic acid, 1-*iso*nicotinyl-2-*iso*propylhydrazine, 2-allyl-1-*iso*nicotinyl-2-*iso*propylhydrazine dihydrochloride and N′-methyl-*iso*nicotinic acid hydrazide.
H. F. W. Kirkpatrick

3106. **The quantitative determination of mepacrine, proguanil and Plazmotsid in tablets.** G. A. Vaisman (*Aptechnoe Delo*, 1953, **2** [6], 25–30).—The powdered tablets are treated with dil. H_2SO_4 and extracted with ether; the methylenedisalicylic acid so obtained is determined bromimetrically. Mepacrine is determined by extracting with water, evaporating the filtered extract to a small vol. and adding excess of 0·1 *N* iodine monochloride in HCl. The ICl consumed is determined by adding KI and back-titrating with $Na_2S_2O_3$. Plazmotsid [the methylenedisalicylic acid salt of 8-(3-diethylaminopropylamino)-6-methoxyquinoline] does not react with ICl under the conditions described. The sum of the proguanil plus mepacrine is determined by evaporating a portion of the aqueous extract, adding ether and titrating with 0·1 *N* NaOH until the aqueous layer is alkaline to phenolphthalein. The results of determinations on various tablets and mixtures are given. E. Hayes

3107. **A contribution to the photometric estimation of acetanilide Lactophenin, phenacetin, Rodilone, Tibatin and Tebethion [thiacetazone] in pharmaceuticals.** J. Richter (*Z. anal. Chem.*, 1954, **142** [4], 277–279).—A rapid method for the colorimetric estimation of the above chemicals in pharmaceuticals is described. One to one hundred mg of any of these is hydrolysed for 5 min. with 12·5 per cent. HCl (20 ml), diluted, nearly neutralised with 15 per cent. NaOH (20 ml) or KOH (25 ml) and diazotised with 0·5 per cent. $NaNO_2$ soln. (5 ml); 0·1 per cent. thymol soln. in 1·5 per cent. NaOH (5 ml) is added, and the orange colour is measured after 2 min. Pyrazolone derivatives and salicylic acid compounds do not interfere. P. S. Stross

3108. **A new technique for performing the Cronheim - Ware reaction.** K. F. Körner (*Z. anal. Chem.*, 1954, **142** [4], 267–275).—In this critical examination of the Cronheim - Ware reaction (*J. Pharmacol. Exp. Ther.*, 1948, **92**, 98) several important modifications are suggested by which the accuracy and reproducibility are much increased. Quartz apparatus suitably treated is found to be essential, and it is found necessary to maintain constant temperature to prevent precipitation of water from the solvent layer; the buffer concentrations have also been changed. The modified method is applied to urine analysis; it was found to be applicable to the determination of strychnine, Pantocain [amethocaine], Dromoran [methorphinan], Cliradon [4-*m*-hydroxyphenyl-1-methyl-4-propionylpiperidine hydrochloride], whilst Cardiazol [leptazol], caffeine, Coramine [nikethamide], Sympatol [1-(*p*-hydroxyphenyl)-2-methylaminoethanol tartrate], Suprifen [1-(*p*-hydroxyphenyl)-2-methylaminopropanol], morphine alkaloids and many pyrazolone derivatives give negative results.
P. S. Stross

3109. **The quantitative determination of Atoxyl [sodium *p*-aminophenylarsonate] by a bromimetric method.** V. A. Gvozdik, M. A. Trotsenko and V. P. Potapova (*Aptechnoe Delo*, 1954, **3** [3], 33–35).—To 10 ml of an aqueous soln. containing about 0·1 g of Atoxyl in a ground-glass flask, add 25 ml of 0·1 *N* $KBrO_3$ soln., 0·5 g of KBr and sufficient conc. HCl to make the final concn. 2 *N*; set aside the stoppered flask in the dark with frequent shaking. Add 20 ml of 0·1 *N* As_2O_3 solution, heat on a water-bath to 60° to 70° C and titrate with 0·1 *N* $KBrO_3$ (indicator, methyl orange). One ml of 0·1 *N* $KBrO_3 \equiv$ 0·005485 g of Atoxyl pentahydrate. The method gives results in agreement with those obtained by the U.S.S.R. Pharmacopoeia VIII method; the limits of error are ± 0·3 per cent. Iodimetric determination of the excess of Br in the first stage gave results less satisfactory than those obtained with the use of As_2O_3. E. Hayes

3110. **The use of flotation in testing pharmaceutical preparations for salts of heavy metals, iron and zinc.** A. G. Belousova (*Aptechnoe Delo*, 1954, **3** [3], 23–27).—The sulphide limit test for lead in pharmaceutical preparations is made more sensitive and reproducible by the addition of a mixture of *iso*butanol and ether (2 + 100) after the sulphide has been precipitated; the ppt. then forms a film at the phase boundary, where it can be more easily observed and compared with a standard. In the $K_4Fe(CN)_6$ test for iron a mixture of 100 vol. of ether, 5 vol. of ethanol and 0·75 vol. of vegetable oil is used to form the non-aqueous phase, and a 12-fold increase in sensitivity is achieved. In the $K_4Fe(CN)_6$ test for zinc, a mixture of *iso*butanol and ether (2 + 100) is used. Semi-micro adaptations of the tests are described. E. Hayes

3111. **The routine determination of the antacid efficiency of aluminium hydroxide gels.** P. R. Clemow and J. W. Lowry (*J. Pharm. Pharmacol.*, 1954, **6** [9], 610–618).—*In vitro* methods for assessing the therapeutic value of antacids are discussed in relation to the routine evaluation of aluminium hydroxide gels. A suitable routine test is as follows. Add quickly 8 g of liquid gel or 0·6 g of dried gel (or an equivalent amount if in any other form) to 250 ml of 0·05 *N* HCl at 37° ± 0·5° C (pH 1·35 to 1·40) in a 250-ml beaker. Agitate the beaker briskly and continuously and determine the pH at intervals. It is suggested that with the dried gel, a pH of ≮ 3·0 should be attained in 20 min. and a pH of ≮ 3·5 in 30 min., and at no time should

the pH exceed 4·0; for the liquid gel, the pH should be $\not<$ 3·5 in 10 min. and at no time $>$ 4·0.
S. C. JOLLY

See also Abstract 2916.

Food

3112. **Rapid method for determining free ammonia in foods.** D. S. Ortiz (*Rev. Soc. Venezol. Quím.*, 1953, **5** [25], 39–46).—To 10 g of the homogenised sample are added 250 ml of H_2O and 20 drops of kerosene. Five ml of aq. NaOH (40 per cent. w/v) are then added and the liberated ammonia is blown over with a current of air into a flask containing 2 ml of H_2SO_4 (0·01 *N*), 100 ml of H_2O and 4 drops of methyl red soln. The time taken for neutralisation of the acid is noted and the NH_3 content of the sample is deduced from tables. L. A. O'NEILL

3113. **Calculation of the integral attenuation index between 400 and 700 millimicrons and of corresponding wavelength for filtered raw-sugar solutions.** F. W. Zerban and L. Sattler (*Anal. Chem.*, 1954, **26** [8], 1363–1365).—An investigation was carried out to determine what single wavelength yields attenuation indices that correlate best with an average obtained from all wavelengths that can contribute to colour within the visible spectrum. From transmittancy data for 97 raw sugars, the curve of attenuation indices was plotted against wavelength for each soln. and averaged over the range 400 to 700 mμ. The wavelength at which the soln. has an attenuation index equal to the average was then read by interpolation. The mean wavelength for all 97 samples was 505·3 mμ, the standard deviation being ± 2·6 per cent. The approx. average measure of the concn. of colouring matter in raw sugars may be determined at 505 mμ.
G. P. COOK

3114. **The determination of fat in starch products, especially those containing milk, by a modified Röse - Gottlieb method.** R. Lehner and A. Estoppey (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [3], 183–185).—The Röse-Gottlieb method gives results 1 to 2 per cent. too low when applied to starch products containing milk. Results are satisfactory by degrading the starch enzymatically before extracting. One g of the starch product is mixed with 0·1 g of takadiastase and 8 ml of water in an extraction tube. The tube is then kept at 60° to 65° C for two hr. with occasional shaking; after this time the starch is completely degraded, as shown by testing with 0·05 *N* iodine soln. To the cooled liquid, 1·5 ml of 25 per cent. aq. NH_3 are added, followed by 10 ml of ethanol; the tube is shaken for 30 sec., 25 ml of ether are added and, after 2 min. vigorous shaking, 25 ml of light petroleum (boiling range 30° to 50°C) are added. The tube is gently shaken for 2 min. and then centrifuged. The non-aqueous layer is siphoned into a tared flask and the fat is determined in the usual way. E. HAYES

3115. **Determination of fat in canned cream soup.** Food Manufacturers' Federation (*Analyst*, 1954, **79**, 509–510).—A method is recommended for determination of fat in canned cream soup. The sample (5 to 10 g) is mixed with 2 ml of ethanol, 10 ml of dil. HCl (25 + 11) are then added and the mixture is heated in a water-bath at 70° to 80° C until the liquid is almost clear. Ethanol (10 ml) is added to the cooled liquid, which is then shaken with 25 ml of ether and re-shaken after addition of 25 ml of light petroleum. The mixed solvent layer is siphoned through a cotton-wool filter into a distillation flask and the aq. layer is re-extracted three times as before with 15-ml portions of each solvent. After removal of the solvent, the residual fat is dried, dissolved in light petroleum and filtered through paper into a tared flask, the solvent is removed and the fat is dried and its purification is repeated until its wt. is constant. A. O. JONES

3116. **Determination of a soluble pectin in apples.** J. M. Lawrence and K. Groves (*J. Agric. Food Chem.*, 1954, **2** [17], 882–885).—Modifications of the Ca pectate method to accelerate the procedure are discussed. A new photometric method is described based on the absorption at 295 mμ of a prepared pectin solution after heating for 15 min. in boiling water with nine volumes of 84 per cent. H_2SO_4 soln. The pectin content of apple juice is taken to represent a soluble pectin level in the apple pulp. Both the above methods were used to estimate the pectin content of samples of juice from apples of various degrees of maturity, and results agreed fairly well with those obtained by measuring evolution of CO_2 in order to determine the polygalacturonic acid content. S.C.I. ABSTR.

3117. **Conductimetric titration of wines.** I. Mareca Cortés (*Inf. Quím. Anal.*, 1954, **8** [3], 86–89, 85).—The determination of sulphate in wine by conductimetric titration with 0·25 *N* Ba acetate is described. D. P. YOUNG

3118. **The determination of acetaldehyde and sulphurous acid in wine and fruit juices by means of the Lieb - Zacherl apparatus.** F. Paul (*Mitt. Wein u.-Obstbau, A*, 1954, **4** [5], 225–234).—The apparatus (originally designed for the determination of lactic acid as acetaldehyde *cf. Hoppe-Seyl. Z.*, 1932, **211**, 211, and 1935, **231**, 88) consists in a boiling-flask, connected through a still-head and a vertical reflux condenser with an absorption-vessel; the still-head carries a side-tube, the capillary end of which dips into the liquid in the flask, so serving for the passing of a current of air or N, and also (for the determination of lactic acid) for the addition of aq. $KMnO_4$ from a separating-funnel. For the determination of aldehyde, the sample (10 to 20 ml, mixed with 2 ml of 5 per cent. aq. H_3PO_4) is gently boiled for 10 to 15 min. in a slow current of air, and the aldehyde is absorbed by a mixture of 2 ml of aq. $NaHSO_3$ (18·9 g of Na_2SO_3 and 4·0 ml of conc. H_2SO_4 per litre) and 10 ml of phosphate buffer of pH 7. The absorption mixture (with washings) is acidified with 1 ml of 10 per cent. HCl (to pH 1 to 2) and the excess of H_2SO_3 is oxidised by careful addition of 0·1 *N* I soln. (starch indicator); after addition of borate buffer soln. (25 ml) to bring the pH to 8·5 to 9, the acetaldehyde - bisulphite is determined iodimetrically. Determinations on standard aldehyde soln. are accurate to within 2 to 3 per cent. The determination of total SO_3'' follows a similar course, except that the sample (10 to 20 ml) is acidified with 5 ml of 25 per cent. H_3PO_4, boiling is conducted under a current of N, and the SO_2 is absorbed either in a borate buffer (pH 9) for iodimetric, or in neutral 1 per cent. H_2O_2 for acidimetric, determination. Combined SO_3'' is determined by titrating a second acidified sample with 0·1 *N* I soln. until a blue coloration is obtained with starch, setting aside with exposure to air until decoloration has occurred, and then proceeding with the distillation. A blank value is obtained by distillation after treating the acidified sample with neutral 1 per cent. H_2O_2 soln. Various minor modifications to the apparatus are discussed. P. S. ARUP

3119. **Chemical standards for coffee powder.** S. Narayanaiyer and K. Ramachandran (*Curr. Sci.*, 1954, **23** [6], 192).—Samples of *Coffea arabica* and *C. robusta* seeds were roasted, powdered and analysed for water extract, alkalinity of ash, caffeine and fat (light petroleum extract) contents, with a view to establishing chemical standards for testing the genuineness of unknown coffee powders. All the samples, except one inferior variety, gave approx. the same value for water extract (26 to 30 per cent.) and alkalinity of ash (19 to 23 per cent.). *C. robusta* has a higher caffeine content (2 to 2·4 per cent.) than the average associated with a seed having a low fat content (7 to 9 per cent.). The figures for *C. arabica* are 1·3 to 1·4 per cent. and 12 to 15 per cent., respectively. A. M. SPRATT

3120. **A new method for the determination of volatile oils in spices and drugs.** H. Hadorn, R. Jungkunz and K. W. Biefer (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [3], 200–221).—Various methods for the determination of volatile oils in spices and drugs are reviewed and some oxidation methods are critically examined. In a proposed new method, water is added to a weighed sample in a small beaker, which is then suspended in a special absorption vessel containing $K_2Cr_2O_7$ - H_2SO_4 and heated in an oven at 103° to 105° C for 1·5 to 2 hr. The oil is volatilised, absorbed by the dichromate soln. and oxidised. The excess of dichromate is determined iodimetrically. The proportion of oil is then calculated with the aid of an empirical factor previously determined for each type of material. Results obtained on various drugs and spices are given. E. HAYES

3121. **Determination of vanillin in vanilla extracts.** A. Maurel and S. Lalement (*Chim. Anal.*, 1954, **36** [9], 241–244).—A colorimetric determination by nitrosation and a titrimetric determination by oximation of vanillin are compared with known gravimetric methods and are shown to be superior to the colorimetric method with Folin - Denis reagent, which is interfered with by impurities present in vanilla extracts. Extracts of vanilla pods in water or water - ethanol are used. In the nitrosation method, a 1 to 5-ml sample of extract is diluted with distilled water, pptd. with Pb acetate, and excess of Pb is removed with 10 per cent. H_2SO_4. The filtrate is made up to a known vol, and, to a 5-ml aliquot buffered with 2 ml acetate buffer to pH 4, are added 2 ml of 10 per cent. aq. $NaNO_2$. The tube is heated for 15 min. in boiling water, and after cooling and making up to 10 ml, the absorption at 415 mμ of the yellow substance formed (presumably 4-hydroxy-3-methoxy-5-nitrosobenzaldehyde) is measured. "Ethyl vanillin" gives an absorption max. at the same wavelength, but *iso*eugenol, anisaldehyde and other phenolic compounds reacting under these conditions have absorption max. at different wavelengths. In the oximation method, 0·5 to 5 ml of the extract is diluted with ethanol and adjusted to pH ≃ 6 with *N* KOH - ethanol (1 to 3 drops), and 5 ml of hydroxyammonium chloride soln. are added. The pH is measured immediately after addition of the reagent, and, after 30 min. when the reaction is complete and all the HCl is liberated from the reacted hydrochloride, the solution is titrated with 0·01 *N* KOH until the pH is the same as at the moment of adding the reagent. A comparison of results is given with Folin - Denis and with two gravimetric methods. E. J. H. BIRCH

3122. **Fluorimetric identification of refined olive oils.** F. Provvedi (*Olii Min.*, 1954, **31** [8], 139–143).—The literature on methods of differentiating between virgin olive oil and refined and solvent-extracted oil by the fluorescence in Wood's light is reviewed. The fluorescence of an oil can be determined in a Beckman spectrophotometer fitted with a fluoroscence attachment; authentic virgin oils examined in this way give low results compared with refined oils. The method permits the detection of refined oil in virgin oil. E. HAYES

3123. **[Determination of] fatty acid composition of castor oil.** J. G. Kane and B. Sreenivasan (*Paint-india*, 1954, **4** [1], 128–131).—The oil is saponified and the potassium soaps are converted directly to methyl esters by treatment with HCl in methanol. Hydroxylated esters are separated from non-hydroxylated ones by treatment with succinic anhydride and extraction of the succinic half-esters with aq. K_2CO_3. From the linoleic ester contents (determined spectrophotometrically) and the iodine values of the total esters and of the two fractions, the complete composition can be calculated. Confirmatory determinations may be made of the saturated ester content (Twitchell method) and the dihydroxystearic ester content (KIO_4 method). The method does not depend on hydroxyl value determinations, which are considered unreliable. A typical fatty acid percentage composition has been found to be: saturated acids, 3·5; oleic acid, 5·5; linoleic acid, 3·5; ricinoleic acid, 85·5; and dihydroxystearic acid, 2. L. A. O'NEILL

3124. **Purification of groundnut oil. IV. Chromatographic separation and estimation of tocopherol in arachis oil.** M. L. Sen Gupta and A. K. Ganguly (*J. Indian Chem. Soc., Ind. News Ed.*, 1953, **16** [4], 175–178).—Interference with the estimation of tocopherol by the Emmerie - Engel reagent caused by presence of peroxides is avoided by preparation of the column with Grade III or IV alumina and light petroleum (boiling range 40° to 60° C), followed by elution with light petroleum (boiling range 60° to 80° C), and development of the colour with a $FeCl_3$ - dipyridyl reagent. M. TADMAN

3125. **Potentiometric titration of antioxidants with ceric sulphate.** F. Wenger (*Mitt. Lebensm. Hyg., Bern*, 1954, **45** [3], 185–200).—Ceric sulphate (0·004 *N*) is used in the titration of various phenolic antioxidants in 72 per cent. ethanol soln., the end-point being determined potentiometrically or by means of a redox indicator. Titration curves are given for 2-*tert.*- and 3-*tert.*-butyl-4-hydroxyanisoles, dihydronorguaiaretic acid, dodecyl, octyl and propyl gallates, guaiacum resin and quinol. At temp. above 50° C, the ethanol is oxidised. Alcoholic extracts of arachis oil and soya-bean oil behave normally, but those of sesame and cotton-seed oils are not satisfactory; olive oil may also contain oxidisable ethanol-soluble substances that interfere. Oxidised oils, especially those with peroxide numbers > 10 are also unsatisfactory. When used as a qualitative test on concentrated extracts, the method permits the detection of as little as 0·003 per cent. of antioxidant in a fat. E. HAYES

3126. **Simplified procedure for extraction and determination of vitamin A in liver.** S. R. Ames, H. A. Risley and P. L. Harris (*Anal. Chem.*, 1954, **26** [8], 1378–1381).—The liver is dried by grinding with anhydrous Na_2SO_4, and the vitamin A is extracted by shaking with ether. The vitamin-A content of an aliquot is determined by the standard

Carr - Price reaction ($SbCl_3$), the resulting blue colour being measured at 620 mμ. The recovery of vitamin A at 3 levels, 30, 150 and 300 μg, averaged 98·6 ± 0·56 per cent., and there was no indication of extraction from the liver of materials that interfere with the determination. The standard deviation of a single result is ± 2·4 per cent. The method compared well with the best of 4 other procedures tried. G. P. Cook

3127. **Paper-chromatographic identification of ascorbic acid and dehydroascorbic acid.** M. Ulmann (*Pharmazie*, 1954, **9** [6], 523–529).—The difficulties of ascorbic acid identification are reviewed and the use of paper-chromatography is described. Preparations of ascorbic acid are chromatographed in a tube in which the paper strip is suspended and through which hydrogen is passed. The solvent used is butanol - acetic acid - water (4:1:5) and an R_F of 0·38 is obtained on Gessner and Kreutzig 3w paper; results for other papers are quoted. The spot is revealed by spraying, usually with $AgNO_3$ solutions (in NH_3 or acetic acid). Oxidation of ascorbic acid is effected by three methods. On bubbling air through 1 per cent. aq. ascorbic acid for 4 hr. and chromatographing, a small amount of dehydroascorbic acid (R_F, 0·44) is produced, but after a week this solution shows a decrease in the dehydroascorbic acid content and an unidentified substance, which reduces $AgNO_3$, (R_F, 0·10) appears as a pale yellow spot on the dried chromatogram. Oxidation with pure oxygen produces similar results. The largest amount of dehydroascorbic acid is obtained by oxidation with quinone (in ether solution). The substance of R_F 0·10 is also obtained. Distinction between ascorbic acid and dehydroascorbic acid on the paper strip may be made by spraying with a solution of 0·3 g of phenylhydrazine and 0·45 g of sodium acetate in 10 ml of water, when only the latter acid forms an osazone. The adaptation of the method to circular paper chromatography is described, the hydrogen atmosphere being applied in a large desiccator. The question of quantitative estimation of the substances on the chromatogram is briefly discussed.
E. J. H. Birch

3128. **Determination of vitamin B_{12} content of feed supplements and effect of pseudo-vitamin B_{12}.** L. Chaiet, T. Miller and A. E. Boley (*J. Agric. Food Chem.*, 1954, **2** [15], 784–786).—A number of commercial (animal protein factor) products were examined for vitamin B_{12} by the isotope dilution method, the U.S.P. *L. leichmannii* titrimetric and turbidimetric methods, the microbiological cup assay method with *Lactobacillus lactis*, Dorner, and with *Escherichia coli*, and two spectrophotometric methods; a few samples were also assayed by the chick-growth method. The microbiological and spectrophotometric methods do not distinguish between vitamin B_{12} and pseudo-vitamin B_{12}; one commercial product was found to contain no vitamin B_{12}, but the microbiologically active ingredient was isolated in the crystalline form and identified as the pseudo-vitamin. The average of the results obtained by the isotope dilution method was lower than that obtained by the U.S.P. microbiological assay. Difficulties were encountered in the spectrophotometric assays and the results are untrustworthy. E. Hayes

3129. **Extraction procedure and determination of the vitamin B_{12} content of some animal livers.** K. G. Shenoy and G. B. Ramasarma (*Arch. Biochem. Biophys.*, 1954, **51** [2], 371–378).—A study is made of various procedures for extracting vitamin B_{12} from liver tissue for subsequent assay by a turbidimetric adaptation of the U.S.P. XIV microbiological assay. The protecting effect of sodium metabisulphite during heat treatment is confirmed, and, by the proposed papain-digestion method, the following average figures for vitamin B_{12} activity (μg per g) were obtained on liver samples: cow, 1·18; buffalo, 1·30; sheep, 1·33; goat, 1·20; pig, 0·59; rabbit, 0·60; chicken, 0·27; rat, 0·052; and mouse, 0·75. These results and the corresponding values for alkali-stable vitamin B_{12} activity are discussed. *Extraction procedure*—Mix 1 g of liver homogenate, 50 ml of water and 0·5 ml of 0·5 per cent. aq. suspension of crude papain in a test tube (3·8 × 20 cm) and heat at 60° C for 1 hr. Add 1 ml of fresh 5 per cent. aq. sodium metabisulphite and steam for 5 min. Cool, dilute to 100 ml and filter through paper. W. H. C. Shaw

See also Abstract 3189.

Sanitation

3130. **Radioactivity assay of water and industrial wastes with internal proportional counter.** L. R. Setter, A. S. Goldin and J. S. Nader (*Anal. Chem.*, 1954, **26** [8], 1304–1306).—Suspended matter in water samples (200 to 1000 ml) is removed by centrifugation or filtration on a cellulose acetate membrane. The filtrate is evaporated and its residue is dried at 103° C on the aluminium counting dish used in the radioactive assay. An internal proportional counter with a U_3O_8 source for determination of operating voltages is used for α- and β-counting. Radiations of 50 μμ curies per litre can be determined within 10 per cent. for α-radiation or 20 per cent. for β-radiation. D. A. Pantony

3131. **Measurement of oxygen uptake by oxidation - reduction potential.** J. P. Horton (*Instruments and Automation*, 1954, **27** [8], 1312–1313).—The rate of metabolisation of bacteria in an activated-sludge system can be determined from potentiometric measurements of redox potential (V_r) in samples of liquor in a stoppered Erlenmeyer flask. The simple apparatus and procedure used at dairy-waste disposal plants of the activated-sludge type are described. By plotting rate of change of V_r against time, hourly variations in metabolic activity can be followed, and the process can be strictly controlled. Temp. and pH of the samples are also recorded. The rate of change of V_r is related directly to the rate of change of dissolved O and to the B.O.D. W. J. Baker

3132. **A comparison of the chemical and biological assays of several strains of pyrethrum flowers.** Colonial Products Advisory Bureau (*Colon. Plant Animal Prod.*, 1954, **4** [1], 59–69).—Reasonable agreement was found between the results of the collaborating laboratories in the U.K. and U.S.A. when fresh and stored flowers of one normal and two high pyrethrin content strains and extracts from these samples were assayed by the A.O.A.C. (seventh edition) method both in its original and modified forms. With the second form pyrethrin I contents were 15 to 20 per cent. higher than with the original method. No consistent differences were found between the toxicities of the 3 strains when tested on a variety of insect species. Biological results indicated a greater loss of toxicity on storage on one high-content strain (C47) than was shown by chemical assay, so current methods of analysis are possibly not suitable for this strain.

which was abnormal in that the ratio of pyrethrin I to pyrethrin II was only about 0·6. S. C. JOLLY

3133. **The assay of γ-hexachlorocyclohexane by chromatography on activated charcoal.** A. Germano, R. Fazan and I. Lossius (*Helv. Chim. Acta*, 1954, **37** [4], 1332–1336).—The reference method of C. V. Bowen (*Anal. Chem.*, 1953, **25**, 42) is improved by using activated charcoal instead of silica gel. The former has the advantage of being easy to prepare and readily available in a uniform quality. Better separations and fewer oily intermediate products are obtained. Quantitative elutions are achieved by mixing it with 20 to 26 per cent. by weight of sucrose. Ether is the most suitable eluting medium for the determination of the γ isomer in a mixture of the α and γ isomers of hexachlorocyclohexanes. As ether would elute γ- and δ-isomers together, light petroleum saturated with nitromethane is used for the separation of a mixture of α-, γ- and δ-isomers. P. S. STROSS

3134. **Determination of the *pp′* isomer of DDT by infra-red spectrometry.** L. Henry, A. Colas and J. Prat (*Chim. et Ind.*, 1954, **71** [5], 919–923).—Technical DDT normally contains 70 to 80 per cent. of the *pp′* isomer, the remainder of the product consisting mainly of the *op′* isomer. The *pp′* and *op′* in the compounds can be determined within $\simeq 2$ per cent. and $\simeq 10$ per cent., respectively, by i.r. spectrometry. The presence of > 1 per cent. of any of the by-products likely to be found in technical DDT is readily detected by examination of the i.r. spectrum of the product, thus obviating the possibility of any serious error arising in the determination of the *pp′* isomer by this method. J. M. JACOBS

3135. **Determination of absolute concentration of deuterium in Thames river water.** G. R. Clarke, W. H. Denton and P. Reynolds (*Nature*, 1954, **174**, 469).—The ratio [HD] to [H_2] for the gas obtained by U reduction of water samples prepared by mixing natural water and pure D_2O in known proportions is determined in a mass spectrometer. The ratio $\Delta C/C_0$ is simply related to the mass spectrographic ratio, where ΔC is the known excess concn. of D in the sample and C_0 is the concn. in the natural water. By plotting $\Delta C/C_0$ against ΔC, a straight line is obtained from which C_0 can be determined. For Thames river water, C_0 is found to be 0·0152 ± 0·0003 per cent., which is greater than the figure for most American fresh waters, and nearly as high as for oceanic waters. This is attributed to Thames water being mainly an initial condensate from atmospheric moisture, and is hence enriched by an amount nearly equal to the original depletion on evaporation from the Atlantic; American river water is by comparison a nearly complete condensate from the depleted atmospheric moisture. H. P. PAGET

See also Abstracts 2956, 2969, 3183.

Agriculture and Plant Biochemistry

3136. **Spectrographic determination of zinc in plant materials.** R. T. Marfori (*Dissert. Abstr.*, 1954, **14** [6], 919–920).—Of various rapid spectrographic methods for determination of Zn in plants the most satisfactory is found to be that in which are used plant ash with a suitable buffer, *e.g.*, Li_2CO_3, and d.c. arc excitation of the zinc line 2138·56 A, with the tellurium line 2142·75 A as a reference line. The max. error from the chemical method is $\simeq 21$ per cent. L. F. TAYLOR

3137. **Analytical studies on the carbohydrates of grasses and clovers. V. Development of a method for the estimation of cell-wall polysaccharides.** V. D. Harwood (*J. Sci. Food Agric.*, 1954, **5** [6], 270–275).—The cell-wall polysaccharides, *viz.*, the carbohydrate fraction of herbage that is insoluble in alcohol and cold water, are determined by a two-stage acid hydrolysis procedure. In the first stage, treatment with *N* H_2SO_4 effects solution and hydrolysis of the greater part of the hemicellulose fraction, with a max. variability in any one sugar of 7 per cent. of the total hydrolysed. The remaining "cellulose" residue is saccharified with 72 per cent. H_2SO_4 during the second stage, with agreement to within 5 per cent. O. M. WHITTON

3138. **Spectrophotometric determination of humulone complex and lupulone in hops.** G. Alderton, G. F. Bailey, J. C. Lewis and F. Stitt (*Anal. Chem.*, 1954, **26** [6], 983–992).—To 5 g of hand-ground hops in a glass-stoppered graduated cylinder, add low-boiling light petroleum to bring the vol. to 100 ml. Shake the liquid by gentle rocking at room temp. for 30 min. and set it aside until the solids settle leaving a clear supernatant liquor. Dilute an aliquot of the clear extract with reagent-grade absolute methanol to a suitable vol. for spectrophotometric reading (for the Beckman DU spectrophotometer dilute 1 to 100 or 200). To the dil. soln. in the absorption cell, add 1 per cent. of its vol. of 0·2 *N* aq. NaOH, mix and read the absorbancies (*A*) immediately at 355, 325 and 275 mμ. Reagent blanks are also run. Calculate results from the equations—

$C_H = -51{\cdot}56\,A_{355} + 73{\cdot}79\,A_{325} - 19{\cdot}07\,A_{275}$,

and $C_L = 55{\cdot}57\,A_{355} - 47{\cdot}59\,A_{325} + 5{\cdot}10\,A_{275}$,

where C_H and C_L are concn. of humulone complex and lupulone, respectively, in mg per litre of soln. measured, and multiply by the dilution factor of the hop sample to find the percentage. This method greatly reduces errors due to background effects. H. F. W. KIRKPATRICK

3139. **Carotene: effect of antioxidant on its determination.** V. H. Booth (*Analyst*, 1954, **79**, 507–509).—The antioxidant NN′-diphenyl-*p*-phenylenediamine (**I**) is used, principally in America, to reduce loss of carotene during storage of lucerne meal. Beauchene *et al.* (*J. Agric. Food Chem.*, 1953, **1**, 461) found that more carotene appeared to be present in lucerne meal immediately after treatment with **I** than in untreated meal. This interference is not observed when light petroleum is used for extraction and Al_2O_3 for the chromatographic separation instead of the mixture of light petroleum and acetone (for extraction) and MgO (for the chromatography) used by Beauchene *et al.* (*loc. cit.*). The interference appears to be due to the chromogenic reaction between MgO and **I** observed by Beauchene *et al.* A. O. JONES

3140. **Analysis of flavones. IV. Application of the zirconium-citric acid test to a spray reaction in paper chromatography.** L. Hörhammer and K. H. Müller (*Arch. Pharm., Berlin*, 1954, **287** [6], 310–313).—In the paper chromatogram, the fluorescence generally persists in those spots that are derived from the more stable five-membered Zr chelates of the flavonols with free hydroxyl groups attached to the C_3 atoms. If the paper chromatogram, after treatment with $ZrOCl_2$ and citric acid, is exposed to HCl vapour, hydrolysis of the glycosides and esters is observed and the resulting flavonols, whose hydroxyl group at the C_3 atom was formerly blocked by sugar or acid residue, show a marked

yellow fluorescence that is not exhibited by those of the flavones. Similar, but less well-defined results are obtained if $Al_2(SO_4)_3$ is used in the formation of the complexes. H. WREN

3141. **Oestrogenic activity of subterranean clover. II. Isolation of genistein from subterranean clover and methods of quantitative estimation.** D. H. Curnow (*Biochem. J.*, 1954, **58** [2], 283–287).—A macro-method for determination of, and a micro-method for detection of, genistein (5:7:4′-trihydroxy*iso*flavone) in plant material are described. In the macro-method, clover (30 g) is extracted with boiling ethanol, and the extract is concentrated *in vacuo*. After adding water (1000 ml) and benzene (4 × 200 ml), the benzene extract is extracted in counter-current fashion with 60 per cent. ethanol. The aq. ethanol phase, which contains > 95 per cent. of the oestrogenic material, is evaporated, and the phenolic material is isolated by extraction with 0·1 *N* NaOH. The phenolic fraction is dissolved in ethanol, and the values for $E^{1\%}_{1\,cm}$ at 231 mμ and $E^{1\%}_{1\,cm}$ at 262·5 mμ are determined spectrophotometrically. The amount of genistein present in the phenolic fraction can be calculated from these values. In the micro-method, a clover leaf is extracted with ethanol, and the same procedure as above is followed. An aliquot of the aq. phase is evaporated, the residue is dissolved in butanol - 1 per cent. HCl, and the solution is transferred to a paper strip. The strips are placed in an atmosphere saturated with the vapour of the aq. phase of a butanol - acetic acid - H_2O mixture (40:10:50, by vol.) for 24 hr. The solvent mixture, butanol - acetic acid - H_2O (40:10:20, by vol.) is then introduced for ascending chromatography. After the solvent front has moved ≃ 30 cm (18 hr.), the strips are dried in a current of air and sprayed with 0·1 per cent. aq. $FeCl_3$. The pink genistein spot is compared with control strips. The content of genistein cannot be determined from the spot length, and only a visual approximation of the content is possible. J. N. ASHLEY

3142. **Determination of functional groups in tannins and lignins. II. Methylation studies.** W. E. Hillis (*J. Soc. Leath. Tr. Chem.*, 1954, **38** [7], 209–214).—The limitations of methylation studies, in which diazomethane is used as an aid to determining the structure of tannins and lignins, are discussed. The effects of methanol, dioxan, dioxan plus 5 per cent. of water, and ether on the yield of methylated derivatives from aromadendrin (dihydrokaempferol) and catechin A have been determined. Methylation with diazomethane results in a change in ether-solubility of lignin. D. LIFF

3143. **Determination of total and assimilable manganese and copper in soils.** C. Bighi and G. Trabanelli (*Ann. Chim., Roma*, 1954, **44** [5–6], 371–379).—Total Cu is obtained by extraction with HCl (sp. gr. 1·18), and assimilable Cu by extraction with aq. KCN buffered to the pH of the soil with acetic acid. Total Mn is obtained by extraction with conc. HNO_3, assimilable Mn by extraction with aq. ammonium acetate and reducible Mn with ammonium acetate plus quinol. Cu is then determined colorimetrically with dithio-oxamide and Mn by oxidation with $(NH_4)_2S_2O_8$. Results for 12 Italian soils are tabulated. L. A. O'NEILL

3144. **Comparison of chemical and bio-assay methods for determination of traces of Chlordane and Heptachlor in food crops.** A. Hartzell, E. E. Storrs and H. P. Burchfield (*Contr. Boyce Thompson Inst.*, 1954, **17** [7], 383–396).—Results by chemical and bio-assay methods agreed closely for samples containing Chlordane, but the bio-assay gave lower results on Heptachlor samples, due to the masking effect of lipids and plant extracts. The chemical methods of analysis were those of Davidow (*Brit. Abstr. C*, 1951, 61) and Polen and Silverman (*Brit. Abstr. C*, 1952, 403). Bio-assay results were obtained by the method of Burchfield.
R. H. HURST

3145. **Direct potentiometric method for chloride ion applied to residues of chlorinated insecticides.** G. K. Helmkamp, F. A. Gunther, J. P. Wolf, III, and J. E. Leonard (*J. Agric. Food Chem.*, 1954, **2** [16], 836–839).—This method depends on the use of a silver - silver chloride *vs.* S.C.E. system. Optimum results are attained at 10 p.p.m. of chloride ($2·82 \times 10^{-4}$ equivalents per litre) with a standard deviation of less than ± 0·1 p.p.m. At concentrations from 0·02 to 1·00 p.p.m. results are reproducible to 0·02 p.p.m. Direct measurements have also been made on solutions containing as much as 10,000 p.p.m. of Cl'. The apparatus is described. A prototype Beckman model GS pH meter was used for the measurement of e.m.f., but a Beckman model H-2 line-operated pH meter was useful within the Cl' range 10 to 1000 p.p.m.; precision was about ± 15 per cent. The method has been applied to chloride determinations in 2000 soil and citrus fruit samples before and after treatment in the field with some 15 insecticides and acaricides, including aldrin, Gammexane, Heptachlor, and Toxaphene.
S.C.I. ABSTR.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

3146. **Automatic apparatus for the determination of nitrogen in organic compounds according to Dumas and Zimmermann.** M. Večeřa (*Chem. Listy*, 1953, **47** [7], 1090–1093).—A refinement of the Dumas - Zimmermann method for micro-determination of N is described. It consists in an automatic electric oven constructed in such a way that combustion temp. of 750° to 760° C is reached during 8 min. The apparatus has been tested on a number of substances with N content between 8 and 22 per cent. G. GLASER

3147. **A rotary scrubber for cleaning large quantities of mercury.** M. Gibson, T. Charnley and S. J. Lord (*J. Sci. Instrum.*, 1954, **31** [9], 345).—Mercury in a suitable container is covered with a cleaning solution. A centrifugal pump constructed from Perspex and driven by a stirrer motor picks up mercury from the bottom of the pool and ejects it as a fine spray in the cleaning solution.
G. SKIRROW

3148. **Apparatus for chromatography and electrophoretic analysis.** C. E. Fox (*Chem. Prod.*, 1954, **17** [7], 260–266).—A short review is given of modern methods and apparatus used in column and paper chromatography. Electrophoresis is also discussed with special reference to the types of apparatus used in vertical and horizontal paper electrophoresis and continuous electrophoresis. G. R. WHALLEY

3149. **A method for continuously registering radioactivity during the course of chromatography.** J. Berthet (*Biochim. Biophys. Acta*, 1954, **15** [1], 1–11).—A recording rate-meter is described suitable

for the measurement of radioactivity during chromatography. Over a range of 300 to 30,000 counts per min. the error is less than 2 per cent., and an automatic selector maintains the instrument on the most suitable of the four scales of sensitivity. A correcting device automatically compensates for the counts lost during the recovery time of the Geiger tube. G. W. CAMBRIDGE

3150. **Apparatus for wet combustion of organic compounds containing carbon-14.** J. G. Burr, jun. (*Anal. Chem.*, 1954, **26** [8], 1395–1396).—The apparatus is an improvement on that described by Neville (*J. Amer. Chem. Soc.*, 1948, **70**, 3501). An easily removable frit on the mercury valve is provided. Use of $SnCl_2.2H_2O$ in the combustion line trap greatly extends the usable period of the mercury valve frit. Increased flexibility is provided by use of ball joints, and an improved form for traps, regulators and bubble counters eliminates possible suck-back of the trap liquids. The radioactivity in the ion chamber is measured as described by Raaen and Ropp (*Anal. Chem.*, 1953, **25**, 174). D. R. GLASSON

3151. **Apparatus for measuring quantitatively the amount of gas in a mixture.** Illinois Testing Laboratories, Inc. (Inventors: A. A. Obermaier and B. A. Ritzenthaler) (Brit. Pat. 708,494. Date Appl. 21.7.52).—The gas is subjected to an electrostatic field between two electrodes and ionised by means of a radium-bearing member. The current flow is compared with a standard established previously for the gas in question, by balancing the current conditions between the electrodes (by adjusting the position of the radium-bearing member) with the current conditions between a pair of electrodes of a standard cell containing the gas to be measured. J. M. JACOBS

3152. **An absorber for mists and gases in air.** A. A. W. Russell (*Analyst*, 1954, **79**, 524–525).—An absorber for mists that are difficult to absorb is described and illustrated. It consists in a cylindrical glass vessel containing an upper sintered-glass disc (B.S. grade I, diam. 6 cm) and a lower one (B.S. grade II), an inlet tube below the lower disc, an exit tube above the upper disc and a stoppered side tube for filling. The absorbing medium is poured in to give a layer $\simeq$ 1 in. deep above the lower disc. The upper disc serves as an impinger and also catches spray. The absorber is suitable for such operations as sampling the air over chromium-plating baths and for absorbing traces of HF in air. The max. flow rate is $\simeq$ 5 litres per min. A. O. JONES

3153. **A four-piece steam distillation apparatus.** M. Antelman (*Bull. A.S.T.M.*, 1954, No. 197, 66).—The simple arrangement described and sketched consists in a separating funnel, a Pyrex-glass distilling flask, a second distilling flask and a condenser. Steam is generated by a slow controlled trickle of H_2O from the funnel on to the hot surface of the Pyrex flask, the steam flowing through the side-arm into the liquid being distilled. W. J. BAKER

3154. **Semi-micro molecular still for quantitative use.** D. F. Rushman and M. G. Simpson (*J. Oil Col. Chem. Ass.*, 1954, **37**, 319–322).—A semi-micro molecular still for the quantitative analysis of $\simeq$ 100-mg samples is described and illustrated. The apparatus is essentially a micro hot-plate in a high vacuum. Material to be analysed is placed in a small metal dish and weighed before it is put on the hotplate. After a given time at a suitable temp. the dish is reweighed. No attempt is made to collect and weigh the distillate directly. Distillation of known mixtures of monomer and dimer esters (mol. wt. $\simeq$ 300 and 600, respectively) shows that recovery of the dimer is good. The apparatus is suitable for use at temp. of the order of 300° C. D. BAILEY

3155. **All-glass adjustable-head liquid reservoir for flow control.** G. J. Janz and E. H. Stevens (*Anal. Chem.*, 1954, **26** [8], 1395).—An all-glass adjustable-head liquid reservoir is designed for delivery of a liquid at a constant-metered rate to a system in which a substantial back-pressure may develop. The principle of a constant-head liquid reservoir is adapted to allow manual adjustment of the head to maintain a constant flow against back-pressure changes. A more reliable relation between flowmeter calibration and liquid input is possible. D. R. GLASSON

3156. **Miniature mixer settler for continuous counter-current solvent extraction.** H. W. Alter, J. W. Codding and A. S. Jennings (*Anal. Chem.*, 1954, **26** [8], 1357–1361).—A miniature mixer settler has been designed as a compact continuous counter-current multi-stage extraction unit to operate with the amounts of material normally handled by laboratory batch counter-current equipment. The contactor has several stages each containing a mixing and a settling section. Every mixing section has an impeller driven by a shaft entering at the top of the stage. Good operation is characterised by steady interfaces in all settling sections and by make-and-break interface-impeller action in the mixing sections. Products may be collected at both ends or removed continuously from any stage. Immersion in a bath for operation at controlled temp. may be arranged. At moderate flow rates with systems having reasonable settling properties, consistent performance at 85 to 95 per cent. efficiency can be expected. D. R. GLASSON

3157. **Temperature measurements in the Mooney viscometer.** G. E. Decker and R. D. Stiehler (*Bull. A.S.T.M.*, 1954, No. 195, 45–51).—Attempts to make reliable measurements of temp. of vulcanised-rubber specimens at 100° C and 145° C in a Mooney viscometer are described. Temp. measured by A.S.T.M. Method D1077–49T (thermocouples inserted through metal plungers) are between those of the rubber in the die cavity and those of the upper die; they are also very sensitive to changes in temp. of the upper die, but independent of the temp. of the lower die. Although the thermal conductivity of insulating materials (hard rubber, resin-bonded glass fibre or nylon) used to support the thermocouple in the die cavity is much smaller than that of a metal plunger, it is still too large for accurate measurement of the rubber temp., and the materials are too fragile. Temp. differentials between die and die cavity were determined in presence and in absence of rubber. The integral die and die-holder of Decker and Roth (*India Rubber World*, 1953, **128**, 339) transfers heat much more rapidly, but reflects more of the temp. gradients in the platens, than the standard two-piece unit. It is concluded that reproducible temp. conditions (within 0·5° F) in the same or different viscometers can be ensured only by measurement and control of the temp. of the upper and lower dies, preferably

by use of a standardised Decker - Roth integral unit. The presence or absence of a rubber grommet in the lower die should be specified. W. J. BAKER

3158. **Adjustment of Mooney viscometer die-closure.** G. E. Decker (*Bull. A.S.T.M.*, 1954, No. 195, 51–52).—By modifying the standard Taylor procedure, the accuracy and reproducibility in die-closure adjustments of a Mooney viscometer are improved considerably. A special cylindrical gauge-block replaces the lower die during the operation of setting the distance between the mating surfaces of the die holders. Since the gauge-block is 0·008 in. thinner than the lower die at the shoulder, adjustment of the die-closure by this method produces the recommended total deformation of 0·004 to 0·006 in. in the various members of the linkage. Gauge-blocks are designed for use with the standard two-piece unit or the Decker - Roth integral unit. W. J. BAKER

3159. **New type of manometer.** F. Tatibouët (*Bull. Soc. Chim. France*, 1954, **21** [6], 811–812).—The apparatus described and illustrated is a combination of the Zimmerli and inclined-arm manometers and enables pressures to be measured continuously during vacuum distillation. The graduated scale slopes at $\simeq 10°$ to facilitate reading pressures of less than 10 mm of mercury; filling with mercury or cleaning liquid (HNO_3 - CrO_3) is effected easily, and the device is put into operation by careful tilting. W. J. BAKER

3160. **Glass and O-ring vacuum valves.** G. W. Preckshot and V. E. Denny (*Anal. Chem.*, 1954, **26** [8], 1398).—Glass O-ring vacuum valves are designed to operate under pressures ranging from high vacuum to about atmospheric. They are particularly useful when liquids are also present and where metals and stopcock and valve greases interfere with experimental results. The O-ring is made of rubber, neoprene, silicone rubber or Teflon. Screw adjustment of the piston movement makes valve operation better. D. R. GLASSON

3161. **Ball valves.** B. B. Bach, J. V. Dawson and L. W. L. Smith (*J. Sci. Instrum.*, 1954, **31** [9], 343).—The end of a standard glass cone with a taper rather longer than the socket (A cone in B socket) is ground to form the seat for a steel ball. The device is suitable for incorporation in glass systems to prevent the passage of mercury. G. SKIRROW

3162. **A sensitive recording extensometer.** G. R. Hyde (*J. Sci. Instrum.*, 1954, **31** [9], 313–314).—Load - extension curves of fibres are obtained within an accuracy of a few per cent. and with a load limit of 250 mg on the instrument described. Load is applied at a constant rate by means of a torsion balance while extension is automatically controlled to keep the balance beam horizontal. A simple recording mechanism permits a continuous plot of the load - extension curve to be made. G. SKIRROW

3163. **Reflux plunger. Automatic liquid joint.** G. Rey-Coquais (*Chim. et Ind.*, 1954, **71** [5], 935–936).—The new type of cooling device described is designed to eliminate ground-glass joints and provide a hermetically sealed liquid joint when such materials as hot conc. mineral acids are used. J. M. JACOBS

See also Abstract 3043.

Optical

3164. **Graphical absorbance-ratio method for rapid two-component spectrophotometric analysis.** R. C. Hirt, F. T. King and R. G. Schmitt (*Anal. Chem.*, 1954, **26** [8], 1270–1273).—The method relies on the ratio of observed absorbances at two selected wavelengths, one being preferably an "isoabsorptive" point, *i.e.*, a point of intersection of the log α against wavelength plots for two spectra. A straight-line plot of the observed absorbances against relative composition as derived from Beer's law is thus permitted for mixtures such as *o*- and *p*- nitrophenols or nitrobenzene and aniline. If no isoabsorptive point exists, a plot of calculated ratio against composition is used. Extension to a three-component system is illustrated by analysis of melamine - ammeline - trimethylolmelamine mixtures. Only three absorptivities are required for a two-component analysis when an isoabsorptive point is used. D. R. GLASSON

3165. **Convenient source-alignment device for a spectrometer.** B. Crawford, jun., R. E. Nightingale, D. L. Rotenburg, E. R. Vincent and H. F. White (*J. Opt. Soc. Amer.*, 1954, **44** [7], 574).—This device, which can be attached permanently to a Perkin-Elmer Model 12 monochromator, enables the intermediate focus of the instrument to be located quickly and accurately. A small tungsten-filament lamp combined with a ground-glass screen acts as test source. The entrance face of the prism is used as a mirror, and the source is permanently located within the spectrometer so that light can be reflected back via the prism face and collimating mirror through the entrance slit along the optic axis. The device can be switched on, even when the spectrometer is recording normally, without appreciable change in the signal. B. S. COOPER

3166. **Double cell for spectral study of interactions between substances.** B. M. Mitzner and S. Z. Lewin (*J. Opt. Soc. Amer.*, 1954, **44** [6], 499–500).—The device described consists essentially in a sandwich of two conventional cells of identical thickness clamped together in a single frame but arranged to be filled independently. For double-beam spectrometry, a double cell in the reference beam has each half filled with one of the two reacting substances, whilst the double cell in the other beam has each half filled with a mixture of the two substances. B. S. COOPER

3167. **A new projection-comparator microphotometer.** J. T. Rozsa and C. G. Moon (*J. Opt. Soc. Amer.*, 1954, **44** [7], 562–565).—The "N.S.L. Spec-Reader" is described. This instrument can be used either as a projection comparator, for viewing at high magnification a small area from each of two spectrum plates, or as a non-recording micro densitometer. For the second application an extra accessory is available to convert the instrument to a recording densitometer particularly suitable for X-ray diffraction films. B. S. COOPER

3168. **A Geiger counter X-ray crystal spectrometer.** P. J. A. McKeown and A. R. Ubbelohde (*J. Sci. Instrum.*, 1954, **31** [9], 321–326).—Circuit details are described of a Geiger - Müller X-ray spectrometer designed for the study of transitions in single crystals. The instrument is capable of measuring shifts of reflections up to high Bragg angles and of giving information about intensity and angle of spread of reflections. The instrument can also be used (with low efficiency and accuracy) for powder specimens. G. SKIRROW

3169. **Accurate analysis with an infra-red double-beam spectrophotometer.** R. Schnurmann and E. Kendrick (*Anal. Chem.*, 1954, **26** [8], 1263–1269).—The effect of instrumental factors on the analytical accuracy of i.r. double-beam spectrophotometers is examined. Xylene - ethylbenzene mixtures can be analysed with an accuracy of $\pm$ 1 per cent. if instrument corrections are applied and at least 4 recordings are averaged. The method of "difference spectra" at fixed wavelengths is demonstrated by measurement of a 0·64 per cent. soln. of *m*-xylene in commercial *p*-xylene. Mean values with a deviation of $\pm$0·1 per cent. are obtained for (60 : 40) *iso*octane - *n*-heptane blends; the single-beam technique is also applicable if the P_0 values are checked and an appreciably larger number of measurements is made than with the double-beam technique. Corrections for finite slit widths must be made. Empirical corrections are preferable to assumptions about distribution of spectra impurity and of response coupled with geometrical approximations to the shape of the absorption band; the tangent to the absorbance - concn. curve in the optimum transmittance region is a closer approximation than the chord drawn through a point on the curve. The technique of difference spectra is speedy and accurate compared with the "cell-in cell-out" method when the spectra of the two samples are very similar. D. R. GLASSON

3170. **Continuous infra-red analysers.** G. E. Smith (*Ind. Eng. Chem.*, 1954, **46** [7], 1376–1377).—The negative filtering-type continuous i.r. analyser is described for both gas and liquid operations. Details of sensitisation, calibration and sensitivity are discussed. Sensitivities attainable in the measurement of water in methanol, propan-2-ol, ethyl methyl ketone, benzene or dichlorodifluoromethane, formaldehyde in water, acetic acid in acetic anhydride or benzene, toluene in benzene, and chlorobenzene in *o*-dichlorobenzene are given. Applications to determinations of chlorobenzene in *o*-dichlorobenzene, acetic acid in acetic anhydride, and toluene in benzene are described, and sample flow rates and other operational variables are discussed. O. M. WHITTON

3171. **A simple infra-red grating spectrometer for use in analysis.** J. Gaunt (*J. Sci. Instrum.*, 1954, **31** [9], 315–318).—Details are given of the design and performance of a cheap i.r. grating spectrometer. Examples of the application to the routine analysis of heavy water are included. G. SKIRROW

3172. **A long-path gas absorption cell.** R. G. Pilston and J. U. White (*J. Opt. Soc. Amer.*, 1954, **44** [7], 572–573).—The proposed cell, which can be evacuated or pressurised, is suitable for the infra-red analysis of components of extremely low concentrations and absorption coefficients. The path length can be varied from 1·25 metres to 10 metres in 1·25-metre steps. B. S. COOPER

3173. **A universal slit-drive mechanism for a single-beam infra-red spectrometer.** D. Chapman and E. T. Sanders (*J. Sci. Instrum.*, 1954, **31** [9], 346).—Details are given of the design of a torque amplifier for an infra-red spectrometer slit. G. SKIRROW

3174. **The L v 2 photocolorimeter.** F. Braun (*Chim. Peintures*, 1954, **17** [6], 178–184).—This photocolorimeter, designed for the measurement of surface colours, is fitted with Polaroids for controlling the intensity of the light, photo-electric cells connected in opposition with a galvanometer to determine when the light intensities of the standard and sample are equal, and three tri-stimulus filters (amber, green and blue). Its use for determining colours according to the C.I.E. and Hunter systems, colour differences and the composition of colour-matching mixtures is described. The formulae quoted for colour-matching are applicable to dyed materials or pale-tinted paints. D. R. DUNCAN

3175. **Simple refractometer cell.** W. G. Young and J. M. Rule (*Anal. Chem.*, 1954, **26** [8], 1393–1394).—The refractometer cell is constructed from a microscopic slide and two square cover glasses resistant to corrosion by organic liquids such as allyl halides. Measurement of refractive indices for liquids having n between 1·30 and 1·48 or 1·54 and 1·70 is normally permitted. Determinations over the complete range are possible if special glass having $n > 1{\cdot}70$ is used. D. R. GLASSON

3176. **Improved hydrogen discharge lamp for use in refractometry.** R. W. King and A. E. Hirschler (*Anal. Chem.*, 1954, **26** [8], 1397–1398).—The lamp described combines a simplification of the design of Campanile and Lantz (see Abstract 3177 below) with a continuous flow method of operation described by Arnold and Donn (*Brit. Abstr. C*, 1948, 70). Water-cooling and a relatively low current source are used in producing a hydrogen spectrum of high purity. Frequent recharging of the discharge tube is avoided by continuous passage of a slow stream of H at a pressure of $\simeq$ 3 mm of mercury. In use with refractometers, the lamp should be placed so that the spiral discharge section is perpendicular to the face of the illuminating prism or to the axis of the mirror on the refractometer. D. R. GLASSON

3177. **Improved hydrogen discharge lamp.** V. A. Campanile and V. Lantz (*Anal. Chem.*, 1954, **26** [8], 1394–1395).—The proposed hydrogen lamp gives an improved source of F and C spectral lines for measurement of refractive dispersion in the analysis of hydrocarbon mixtures. The lamp incorporates a convenient means for charging with H, based on the passage of the gas through Pd at red heat. D. R. GLASSON

3178. **Becker value of manila rope by photo-electric reflectometry.** S. B. Newman, H. K. Hammond, III, and H. F. Riddell (*Bull. A.S.T.M.*, 1954, No. 199, 84–86).—The proposed direct-reading photo-electric reflectometer for evaluating the quality of cordage by colour has special filters. Results are obtained more rapidly and are more accurate than by the standard visual method; the systematic variation between the two methods is $\pm$ 0·3 unit over the range of Becker values 35 to 55. W. J. BAKER

3179. **Instruments for measuring gloss, hiding power and colour of paint films.** D. B. Judd (*Amer. Paint J.*, 1954, **38** [41], 66–68, 70, 72, 74, 76).—The fundamental principles on which these measurements are based are discussed in detail; the instruments used are considered more briefly. A bibliography of 14 references is appended. D. R. DUNCAN

3180. **A method for the electrolytic etching of aluminium for microscopic examination.** P. A. Raine, H. J. Ellis and L. W. Terry (*Metallurgia*, 1954, **50**, 45–46, 52).—An electrolytic etching method devised for the examination of sections of

extruded aluminium tube is described. The method is designed to indicate: (*i*) grain size and structure; (*ii*) transcrystalline flow lines characteristic of the extrusion cycle; and (*iii*) intercrystalline defects (voids, laminations and inclusions) indicative of faulty extrusion. The method can be used for other types of sample, *e.g.*, flat strip.

G. C. JONES

Thermal

3181. **A cryostat.** E. P. Hall and W. L. Nelson (*Anal. Chem.*, 1954, **26** [8], 1393).—The cryostat described enables maintenance of a thermocouple reference junction at 0° C over several days; equilibrium is maintained as the rate of heat influx to the ice-water cell is low (< 70 cal. per day). Insulation and maintenance as stated prolong the life of the cell to ≃ 20 days. D. R. GLASSON

Electrical

3182. **A simplified circuit and conductimetric tube for chemical analysis at low-frequency.** G. G. Blake (*Electron. Eng.*, 1954, **26**, 316–317).—An iron-cored choke and capacitors replace the transformer in a conductimetric circuit for chemical analysis. A special conductimetric tube is described which replaces the conductivity cell and which may be immersed in any quantity of solution.

G. SKIRROW

3183. **Continuous recording of concentration of organic matter in waste water.** R. Kieselbach (*Anal. Chem.*, 1954, **26** [8], 1312–1318).—A method of continuous determination of dissolved organic matter in a stream of waste water is described. Direct recording of concn. in p.p.m. by wt. of organic carbon is possible, changes of 1 p.p.m. being detectable. Initial response to a change in sample composition occurs in 10 min., 67 per cent. response being attained in an additional 4 min. The process involves filtration of the sample, pptn. of carbonates, oxidation of organic carbon by chromic acid at 250° C, stripping, purification and ultimate determination of the resulting CO_2. Powdered antimony and Drierite remove chlorine and water vapour. Thermal conductivity analysis of CO_2 is practicable; 200 p.p.m. of organic carbon produce 1 per cent. by vol. of CO_2 in the oxygen stripping gas. With a Gow-Mac M/T-T-8 thermal conductivity cell with refinements, the necessary 1·5 mV output for this concn. is attainable. A record of the analysis of a typical plant waste stream is presented and development of a fully automatic analyser is outlined. D. R. GLASSON

3184. **Automatic recorder for continuous determination of oxygen in gases using the dropping-mercury electrode.** T. B. Larchar, sen., and M. Czuha, jun. (*Anal. Chem.*, 1954, **26** [8], 1351–1354).—A continuous automatic analysing system is developed for determining the oxygen content of the exit gases in nitrogen purification units or hydrocarbon feeds in reactors used for synthetic-rubber production. Errors found in periodic sampling procedures are eliminated by its use.

D. R. GLASSON

3185. **An automatic differential potentiometric titrator.** H. V. Malmstadt and E. R. Fett (*Anal. Chem.*, 1954, **26** [8], 1348–1351).—The simple and inexpensive automatic differential potentiometric titrator described does not require any instrument adjustments before a titration. The end-point potential need not be known or set, as the electronic circuit computes the second derivative voltage of the ordinary potentiometric curve and this voltage is ideally suited to trigger a relay system, which turns the burette off at the inflection point (end-point) of the titration. Excellent precision and accuracy are given for the two redox systems Fe^{++} - dichromate, Fe^{++} - Ce^{+++} used in testing the titrator.

D. R. GLASSON

3186. **A simple quinhydrone pH meter.** I. Levin (*Chemist Analyst*, 1954, **43** [3], 74–76).—The advantages and limitations of the quinhydrone electrode are described, and particulars are given for the construction of a pH meter in which quinhydrone and calomel half-cells are connected by an agar-saturated KCl soln. bridge operated by a 1·5 V dry cell. A microammeter with a range of 0 to 500 μA enables pH to be estimated to 0·02 unit.

H. P. PAGET

3187. **Precise pH measurements with the high-resistance glass electrode.** R. M. Engelbrecht (*Dissert. Abstr.*, 1954, **14** [6], 925–926).—An accuracy of more than ± 0·01 pH units is reported for pH measurement with the high-resistance glass electrode.

L. F. TAYLOR

3188. **An improved technique for the micro-electrophoresis of oil drops.** D. A. Smith (*Brit. J. Appl. Phys.*, 1954, **5** [9], 325–328).—The "steam-jet" method of preparing hydrocarbon dispersions has been modified to reduce atmospheric contamination. Measurements made in a modified micro-electrophoresis cell between 2½ and 7 hr. after preparation of the dispersion with NaCl or sodium pyroborate solutions as dispersion medium gave for the mobilities of *cyclo*hexane and *n*-hexane, respectively, at 24·9° C 2·94 and 3·52 μ per sec. per V per cm at pH 9·0 and 3·22 and 3·90 μ per sec. per V per cm at pH 11, when Na^+ concn. was 0·01 *N*. In 0·1 *N* solutions containing Na^+ and at a pH of 9·0, the mobility of *cyclo*hexane was 2·69 μ per sec. per V per cm.

G. SKIRROW

3189. **A simple apparatus and technique for preparative paper electrophoresis.** J. R. Cannon and A. R. Gilson (*Chem. & Ind.*, 1954, [5], 120–121).—A Perspex trough is supported beneath a Perspex cover that fits over a large glass jar. Along the bottom of the trough and jar are platinum wire electrodes. A thin even starting line of test solution is applied to both sides of seed test paper, which is then wetted with electrolyte to within 1 cm of this line and suspended from studs inside the trough so as to hang down vertically into the glass jar. Both trough and jar contain electrolyte. A voltage gradient of ≃ 3·9 V per cm is found most satisfactory, but higher voltages distort the bands. With gram quantities of various mixtures of the cobalt-containing hydrolysis products of vitamin B_{12}, satisfactory separation is attained after ≃ 12 hr.

D. LIFF

3190. **Automatic mass-spectrometric analysis.** B. F. Dudenbostel, jun., and W. Priestley, jun. (*Anal. Chem.*, 1954, **26** [8], 1275–1278).—In a rapid method of obtaining mass spectrometer analyses, the application of an automatic peak selector (Spectro-Sadic) and an "analogue-to-digital" converter permits direct tabulation of mass spectrometer ion current pre-selected analytical peaks. These data, furnished automatically by an electric typewriter or on punched cards or tape are then fed to a high-speed computer for calculation and tabulation (IBM summary punch and card programmed calculator). D. R. GLASSON

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
atomic	at.
boiling-point	b.p.
British thermal unit	B.Th.U.
calculated	(calc.)
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
critical	crit.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment, -al	expt.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
indefinite	indef.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim -um, -a	max.
melting-point	m.p.
microgram	μg
micro-litre	μl
micron	μ
milliampere	mA
milligram	mg
millilitre	ml
millimetre	mm
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
organic	org.
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	$n\lambda^t$
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]\lambda^t$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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ANALYTICAL ABSTRACTS

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